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Project HORIZONTAL Validation Report on polychlorinated dibenzodioxins, polychlorinated dibenzofurans and dioxin-like polychlorinated biphenyls

Validation of a horizontal standard for the determination of polychlorinated dibenzodioxins (PCDD), polychlorinated dibenzofurans (PCDF) and dioxins-like polychlorinated biphenyls (DL PCB) in soils, sludges and treated biowaste using high resolution mass selective detection (HR GC-MS) in a European Intercomparison Exercise

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Summary

Project HORIZONTAL is interdisciplinary aiming at a harmonisation and horizontal standardisation of test procedures, in particular for sludge, soils and biowastes. In the context of this standardization project, a series of draft technical specification were designed upon an extensive desk study, fine-tuned after expert consultations and finally validated in international intercomparisons exercise.

This report summarises the work performed within the validation study of the draft standard for the determination of polychlorinated dibenzodioxins (PCDD), polychlorinated dibenzofurans (PCDF) and dioxins-like polychlorinated biphenyls (DL PCB) in soils, sludge and treated bio-waste using high resolution mass selective detection (HR GC-MS). It further explains the underlying statistical concept for the calculation of reproducibility and repeatability from intercomparisons data. In addition all single values, results of the statistical evaluation as well as background information on the validation materials used are described and explained.

Abbreviations

Throughout this report the following abbreviations are used:

ANOVA	Analysis of variances
CAS	Chemical Abstracts System
CEN	Comitteé Européen de Normalisation
DG	Directorate General
DL PCB	Dioxins-like Polychlorinated Biphenyls
ECD	Electron Capture Detector
ECN	Energy Research Centre for the Netherlands
EU	European Union
HR GC-MS	High Resolution Mass Selective Detection
IES	Institute for Environment and Sustainability
IT	Information Technology
ISO	International Organization for Standardisation
JRC	Joint Research Centre
MILC	Measure Interlaboratory Comparison
p	Number of labs
PCDD	Polychlorinated Dibenzodioxins
PCDF	Polychlorinated Dibnzofurans
r	Repeatability limit
R	Reproducibility limit
s_r	Repeatability standard deviation
s_R	Reproducibility standard deviation
TC	Technical Committee

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Introduction to the validation project

Project HORIZONTAL is interdisciplinary aiming at a harmonisation and horizontal standardisation of test procedures, in particular for sludge, soils and biowastes. It was created as in response to the European Commission Mandate M 330 given to CEN, asking for the development and validation of those standards in support of forthcoming EU Directives, such as:

- The revision of the Sewage Sludge Directive 86/278/EEC.
- The Directive on the biological treatment of biodegradable waste.
- The initiative on a legal framework for soil monitoring in Europe.

This mandate explicitly considers standards for the entire analytical procedure (i.e., sampling, pre-treatment and analytical measurement methods for inorganic, organic, hygiene and biological parameters). These are grouped into classes according to their physical/chemical properties, which in turn determine the methods needed to quantify the potential impact on human and animal health, plant uptake, soil function and groundwater quality. As the materials generally feature a mixture of different types of contaminants, it is important to provide an integrated answer covering evaluation of all relevant pollutants.

In order to fulfil the requirements of the aforementioned mandate, the European Commissions Joint Research Centre (JRC) and its Directorate-General for Environment (DG ENV) together with the Technical Committees of the European Standardisation Committee (CEN TCs) concerned designed a pre-normative research initiative called Project HORIZONTAL and presented it to the Commission and the Environmental Authorities in the Member States.

After an extensive literature research and careful evaluation of the feasibility of a given horizontal standard, the standards were drafted and finally validated in a European laboratory intercomparison.

The underlying statistical concept, information about the materials used, details about the participants, measurement results obtained as well as the derived performance characteristics obtained for the determination of polychlorinated dibenzodioxins (PCDD), polychlorinated dibenzofurans (PCDF) and dioxins-like polychlorinated biphenyls (DL PCB) are described hereafter.

1.1 Statistical concept underlying the validation

According to the requirements of the work package concerning data handling & interpretation of the project HORIZONTAL-ORG the respective validation intercomparisons have to be evaluated according to the principles laid down in ISO standard 5725-2:1994. In particular repeatability and reproducibility of the draft standard methods have to be determined. The determination of trueness would require the availability of independent reference values for the materials investigated. This, however, is not possible and was not requested in the frame of this work. In the following, the approach chosen is explained.

1.1.1 Introduction to the statistical model

The statistical model used in ISO 5725 for estimation of accuracy of a measurement method assumes that every test result is the sum of three components:

$$y = m + B + e$$

y: test result

m: general mean

B: laboratory component of bias under repeatability conditions

e: random error occurring in every measurement under repeatability conditions

In the workprogram the quantification of term *e* is explicitly asked for (i.e. repeatability and reproducibility). The repeatability variance is measured directly as the variance of the error term *e*, but the reproducibility depends on the sum of the repeatability variance and the between-laboratory variance:

$$\sigma_r = \sqrt{\overline{\mathrm{var}(e)}}$$

$$\sigma_R = \sqrt{\sigma_L^2 + \sigma_r^2} \qquad \text{with} \qquad \sigma_L = \sqrt{\mathrm{var}(B)}$$

However, soil, biowaste and sludge are multi-phase materials, i.e. they contain two or more distinct types of particles which are fundamentally different in their properties and composition. As a consequence, this introduces an important source of variation for the intercomparison exercise which needs to be considered, i.e. the inherent heterogeneity of the materials.

Thus, a contribution of variation between samples *H* is introduced to the general statistical model:

$$y = m + B + e + H$$

Using ANOVA techniques the different variances are calculated and separated for the evaluation.

1.1.2 Requirements for precision experiment

Layout of the experiment

A suite of 10 to 12 different materials (soil, sludge and biowaste) has been made available for the intercomparison exercise. For each parameter investigated, at least 10 to laboratories should be nominated to participate. The same laboratories should be used for different parameters as far as possible. Due to the complexity of analysis and the respective workload to the laboratories, it was decided to propose three materials for the validation of the PCDD, PCDF and DL PCB draft standard.

Each laboratory received two bottles of each material and was be requested to perform 4 independent analysis per material[1] (2 per bottle) using the respective draft standard methods. The 4 analysis per material should be carried out under repeatability conditions (i.e. same operator[2], same equipment, within a short period of time). As far as possible, also the different materials should be measured under repeatability conditions; however, changes of e.g. operator or equipment are permitted, but must be reported. Likewise, different materials can be analysed on different days if necessary.

Equipment used in the experiment needed to be checked prior to the experiment according to the requirements of the draft standard. The results of these checks have to be documented. Similarly, date and time of each measurement had to be recorded for verification of repeatability conditions.

An appropriate timeframe for the entire exercise has been set and was to be respected.

Recruitment of the laboratories

Each sub-workpackage leader of HORIZONTAL was asked to select the laboratories using the information from section 5.2 of ISO 5725-2:1994 and provide the signed questionnaires (see also Annex 1). The workpackage leaders were responsible for providing the laboratories with the draft standard method and explaining the context of this exercise.

Preparation and use of the materials

Materials used for the exercise were prepared according to the general requirements for reference materials as laid down in ISO Guide 34. Materials were accompanied by instructions for use.

Reporting of results

Online submissions of results using an internet-based IT platform as well as XLS-Spreadsheets were used. In case of online data submission, the participating laboratories received a unique and confidential login and password in due time, enabling them to

[1] Independent analysis means analysis of independent test portions, applying the entire analytical scheme to this test portion, from e.g. extraction to quantification. For instance it does not mean replicate injections of aliquots into a GC-MS instrument.

[2] Operator in this context may also consist of a fixed team of persons, e.g. one person performing extraction, one clean-up, one quantification.

enter their data in a structured form. For authentication purposes a signed printout had to be submitted by mail.

The online data submission included a detailed questionnaire for additional information on the measurements.

1.1.3 Statistical analysis

Statistical analysis of data followed the requirements of ISO 5725-2:1994 and ISO 5725-5:1998. Appropriate tests for the homogeneity of variance, detection of outliers and normal distribution were applied. Statistical evaluation was done using an Excel Macro, developed, tested and successfully applied in other occasion by ECN. Evaluation was executed jointly by JRC and ECN.

1.2 Validation exercise for PCDD, PCDF and DL PCB

1.2.1 Isomers to be measured

The following PCDD isomers were selected for the validation exercise: 2,3,7,8,-TCDD; 1,2,3,7,8-PeCDD; 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDD; 1,2,3,7,8,9-HxCDD; 1,2,3,4,6,7,8-HpCDD; OCDD.

The following PCDF isomers were selected for the validation exercise: 2,3,7,8,-TCDF; 1,2,3,7,8-PeCDF; 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDF; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8,9-HxCDF; 2,3,4,6,7,8-HxCDF; 1,2,3,4,6,7,8-HpCDF; 1,2,3,4,7,8,9-HpCDF; OCDF;

The following DL PCB congeners were selected for the validation exercise:

PCB-77, PCB-81, PCB-105, PCB-114, PCB-118, PCB-123, PCB-126, PCB-156, PCB-157, PCB-167, PCB-169, PCB-189

1.2.2 Samples dispatched for the validation of PCDD, PCDF and DL PCB

After a preliminary rough screening, the following materials were used for the validation round of PCDD, PCDF and DL PCB.

- Compost 1 A pollutant loaded compost material from Vienna
- Sewage Sludge 1 A mixed sewage sludge from Essen
- Soil 3 A sludge amended soil from Barcelona

A more detailed description of background concentrations can be found in Annex 2 to this report. The samples were dispatched simultaneously to all participants using a private courier service.

1.2.3 Draft standards to be followed

The draft standards to be followed could be downloaded following this link, which is situated on the website of the Project HORIZONTAL:

http://www.ecn.nl/docs/society/horizontal/Dioxin_Furan_DL-PCB_Standard_for_validation.pdf

1.2.4 Analytical program

Of each of the three materials 2 bottles had to be analyzed and each bottle had to analyze independently twice. As mentioned above analyses were to be done under repeatability conditions. Results were to be reported referring to DRY MATTER content. The choice, how to apply d.m. correction was free for each participant.

1.2.5 Timing and Submission of data

Dispatch of samples was done on the 18th of October 2006. For users of the Online data submission system (MILC), User Registration was possible from 14th of November 2006 with opening of the MILC Data Submission on 1st of December 2006. The deadline for submission of results has been set for PCDD, PCDF and DL PCB to the 12th of January 2007, but was extended to the end of the same months. After that no further submission was possible.

Alternatively the participants were allowed to submit data electronically as Excel sheet using simply Email.

All data were treated in a confidential way. Any presentation hereafter will refer only to numerical data and it will not be possible to identify the originating laboratory. Lab Codes displayed are NOT related to the order of laboratories hereafter.

In addition to the information provide a Helpdesk was implemented in order to give quick and individual response to the participants during and immediately after the validation study. In case of doubt and suspected transcription errors, further enquires were conducted by JRC.

1.2.6 Participants

The following table lists the participating organizations and entities in the validation exercise for the horizontal PCDD, PCDF and DL PCB standard;

- Austria
  - Umweltbundesamt
- Belgium
  - VITO
- Finland
  - Consulting Engineers Paavo Ristola Ltd.
- France
  - INRA
- Italy
  - European Commission DG JRC IES
- Sweden
  - ALCONTROL AB

1.3 Summary results and derived performance characteristics

The result of the various statistical evaluation including outlier tests, calculation of repeatability and reproducibility standard deviation for the congeners of interest can be found in Annex 3 of this report. In addition, all data submitted by the participants as well as those considered for the calculation of the performance characteristics are listed in Annex 3 to this report.

Based on these calculations the following results were obtained in the validation round upon statistical evaluation according to ISO 5725-2. The average values, the repeatability standard deviation (s_r) and the reproducibility standard deviation (s_R) were obtained (Table1).

The repeatability is determined as an interval around a measurement result (i.e. "repeatability limit"). This interval corresponds to the maximum difference that can be expected (with a 95% statistical confidence) between one test result and another, both test results being obtained under the following conditions: The tests are performed in accordance with all the requirements of the present standard by the same laboratory using its own facilities and testing laboratory samples obtained from the same primary field sample and prepared under identical procedures. The repeatability limit was calculated using the relationship: $r_{test} = f \cdot \sqrt{2} \cdot s_{r,test}$ with the critical range factor f = 2.

The reproducibility, like repeatability is also determined as an interval around a measurement result (i.e. "reproducibility limit"). This interval corresponds to the maximum difference that can be expected (with a 95% statistical confidence) between one test result and another test result obtained by another laboratory, both test results being obtained under the following conditions : The tests are performed in accordance with all the requirements of the present standard by two different laboratories using their own facilities and testing laboratory samples obtained from the same primary field sample and prepared under identical procedures. The reproducibility limit was calculated using the relationship: $R = f \cdot \sqrt{2} \cdot s_R$ with the critical range factor f = 2.

Table 1 - Results of the interlaboratory comparison studies of the determination of PCDD, PCDF and DL PCB by HR GC-MS in treated biowaste, sludge and soil. All concentrations are expressed in ug/kg dm.

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data	Number of data reported below detection	Total no of data
Sludge 1	DL PCB 77	6.666	5.01%	11.7%	0.94	2.18	5	0	19	0	19
Compost 1	DL PCB 77	0.132	7.01%	11.7%	0.03	0.04	4	0	16	0	16
Soil 3	DL PCB 77	*0.0258*		*17.4*			3		11	8	19
Sludge 1	DL PCB 81	0.524	10.52%	48.1%	0.15	0.71	5	0	19	0	19
Compost 1	DL PCB 81	*0.0190*		*79.7*			2		8	8	16
Soil 3	DL PCB 81	*0.0022*		*66.5*			3		8	8	16
Sludge 1	DL PCB 105	19.184	7.07%	14.7%	3.80	7.92	4	1	16	0	18
Compost 1	DL PCB 105	0.683	9.19%	19.4%	0.18	0.37	4	0	16	0	16
Soil 3	DL PCB 105	0.096	5.47%	12.3%	0.01	0.03	4	0	15	4	19
Sludge 1	DL PCB 114	1.369	4.91%	28.8%	0.19	1.10	4	1	16	0	20
Compost 1	DL PCB 114	*0.0504*		*60.2*			2		8	8	16
Soil 3	DL PCB 114	*0.0255*		*154.1*			3		10	8	18
Sludge 1	DL PCB 118	32.585	2.59%	12.0%	2.37	10.92	4	1	16	0	20
Compost 1	DL PCB 118	1.945	9.74%	15.6%	0.53	0.85	4	0	16	0	16
Soil 3	DL PCB 118	0.218	7.46%	19.5%	0.05	0.12	5	0	19	0	19
Sludge 1	DL PCB 123	*1.9351*		*89.0*			5		20	0	20
Compost 1	DL PCB 123	*0.1192*		*84.8*			2		12	4	16
Soil 3	DL PCB 123	*0.0081*		*37.8*			3		8	11	19

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data	Number of data reported below detection	Total no of data
Sludge 1	DL PCB 126	0.250	11.41%	33.0%	0.08	0.23	5	0	19	0	19
Compost 1	DL PCB 126	*0.0359*		*17.8*			2		8	8	16
Soil 3	DL PCB 126	*0.0024*		*35.5*			3		10	9	19
Sludge1	DL PCB 156	7.310	3.54%	10.2%	0.72	2.09	4	1	15	1	20
Compost 1	DL PCB 156	0.882	27.30%	28.7%	0.67	0.71	4	0	16	0	16
Soil 3	DL PCB 156	*0.0453*		*12.6*			3		12	7	19
Sludge 1	DL PCB 157	0.864	6.54%	19.3%	0.16	0.47	4	1	16	0	20
Compost 1	DL PCB 157	0.102	17.20%	21.7%	0.05	0.06	4	0	16	0	16
Soil 3	DL PCB 157	*0.0079*		*18.2*			3		8	11	19
Sludge 1	DL PCB 167	*4.0002*		*51.8*			5		20	0	20
Compost 1	DL PCB 167	*0.7498*		*96.5*			2		16	0	16
Soil 3	DL PCB 167	*0.0195*		*16.3*			3		8	11	19
Sludge 1	DL PCB 169	*0.0415*		*81.9*			3		12	6	18
Compost 1	DL PCB 169	*0.0034*		*17.4*			2		8	8	16
Soil 3	DL PCB 169	*0.0122*		*254.8*			3		7	9	16
Sludge 1	DL PCB 189	*1.1565*		*67.2*			5		20	0	20
Compost 1	DL PCB 189	0.177	37.36%	37.4%	0.18	0.18	4	0	16	0	16
Soil 3	DL PCB 189	*0.0076*		*14.7*			3		8	11	19
Sludge 1	Total DL PCB	75.639	1.92%	13.5%	4.07	28.52	4	1	15		
Compost 1	Total DL PCB	4.664	17.71%	24.3%	2.31	3.18	4	0	16		
Soil 3	Total DL PCB	0.370	11.94%	18.9%	0.12	0.20	5	0	19		

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data	Number of data reported below detection	Total no of data
Sludge 1	1,2,3,4,6,7,8-HpCDD	185.260	11.65%	20.0%	60.44	103.75	6	0	24	4	28
Compost 1	1,2,3,4,6,7,8-HpCDD	175.856	7.85%	40.7%	38.65	200.49	5	0	20	0	20
Soil 3	1,2,3,4,6,7,8-HpCDD	6.538	8.15%	18.8%	1.49	3.45	4	2	15	0	23
Sludge 1	1,2,3,4,7,8-HxCDD	3.498	6.90%	14.1%	0.68	1.38	4	1	15	5	24
Compost 1	1,2,3,4,7,8-HxCDD	*0.74*		*17.6*			2		8	12	20
Soil 3	1,2,3,4,7,8-HxCDD	*0.07*		*41.9*			2		5	18	23
Sludge 1	1,2,3,6,7,8-HxCDD									24	24
Compost 1	1,2,3,6,7,8-HxCDD	*3.61*		*23.3*			3		12	8	20
Soil 3	1,2,3,6,7,8-HxCDD	*0.33*		*26.7*			3		5	18	23
Sludge 1	1,2,3,7,8,9-HxCDD	6.427	14.43%	48.7%	2.60	8.77	4	1	16	4	24
Compost 1	1,2,3,7,8,9-HxCDD	2.086	9.86%	30.1%	0.58	1.76	4	0	14	6	20
Soil 3	1,2,3,7,8,9-HxCDD	*0.43*		*174.0*			3		5	18	23

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data	Number of data reported below detection	Total no of data
Sludge 1	1,2,3,7,8-PeCDD	2.285	18.09%	54.0%	1.16	3.46	4	0	16	8	24
Compost 1	1,2,3,7,8-PeCDD	*0.54*		*12.9*			2		8	12	20
Soil 3	1,2,3,7,8-PeCDD	*0.10*		*15.2*			1		4	19	23
Sludge 1	OCDD	1478	13.83%	22.8%	572.63	942.60	6	0	24	0	24
Compost 1	OCDD	1031	11.19%	42.9%	323.00	1238.75	5	0	20	0	20
Soil 3	OCDD	56.9	5.10%	24.7%	8.13	39.34	5	1	19	0	23
Sludge 1	2,3,7,8-TCDD	*0.77*		*32.9*			3		9	15	24
Compost 1	2,3,7,8-TCDD	*0.17*		*12.4*			1		4	16	20
Soil 3	2,3,7,8-TCDD	*0.55*		*74.8*			1		2	22	24
Sludge 1	OCDF	196.966	13.90%	22.0%	76.68	121.33	6	0	24	0	24
Compost 1	OCDF	26.395	24.25%	27.6%	17.92	20.41	4	1	16	0	20
Soil 3	OCDF	42.273	9.34%	21.4%	11.06	25.37	5	1	19	0	23
Sludge 1	1,2,3,4,6,7,8-HpCDF	95.411	8.97%	19.2%	23.96	51.19	6	0	24	0	24
Compost 1	1,2,3,4,6,7,8-HpCDF						5		20	0	20
Soil 3	1,2,3,4,6,7,8-HpCDF	12.424	20.04%	21.6%	6.97	7.52	6	0	23	0	23
Sludge 1	1,2,3,4,7,8,9-HpCDF	7.407	23.63%	34.7%	4.90	7.20	5	0	20	4	24
Compost 1	1,2,3,4,7,8,9-HpCDF	*1.98*		*73.9*			4		12	8	20
Soil 3	1,2,3,4,7,8,9-HpCDF	*0.21*		*21.8*			2		6	17	23

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data	Number of data reported below detection	Total no of data
Sludge 1	1,2,3,4,7,8-HxCDF	*14.09*		*66.3*			5	0	20	4	24
Compost 1	1,2,3,4,7,8-HxCDF	*2.42*		*41.1*			4		16	4	20
Soil 3	1,2,3,4,7,8-HxCDF	*0.46*		*165.1*			2		10	13	23
Sludge 1	1,2,3,7,8,9-HxCDF	*2.32*		*82.0*			4		15	9	24
Compost 1	1,2,3,7,8,9-HxCDF	*0.22*		*36.1*			4		6	14	20
Soil 3	1,2,3,7,8,9-HxCDF	*0.05*		*47.3*			2		4	19	23
Sludge 1	1,2,3,6,7,8-HxCDF	7.397	10.87%	21.3%	2.25	4.40	5	0	20	4	24
Compost 1	1,2,3,6,7,8-HxCDF	*2.50*		*93.1*			4		13	7	20
Soil 3	1,2,3,6,7,8-HxCDF	*0.19*		*18.0*			2		7	16	23
Sludge 1	2,3,4,6,7,8-HxCDF	*9.64*		*25.6*			5		19	5	24
Compost 1	2,3,4,6,7,8-HxCDF	*2.35*		*40.6*			5		15	5	20
Soil 3	2,3,4,6,7,8-HxCDF	*1.99*		*222.5*			2		11	12	23

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data	Number of data reported below detection	Total no of data
Sludge 1	1,2,3,7,8-PeCDF	5.203	8.32%	23.8%	1.21	3.47	4	0	15	9	24
Compost 1	1,2,3,7,8-PeCDF	1.49		35.5			4		12	8	20
Soil 3	1,2,3,7,8-PeCDF	0.17		9.9			2		5	18	23
Sludge 1	2,3,4,7,8-PeCDF	9.784	31.88%	33.3%	8.73	9.12	5	0	20	4	24
Compost 1	2,3,4,7,8-PeCDF	*2.10*		*36.1*			4		14	6	20
Soil 3	2,3,4,7,8-PeCDF	*1.24*		*47.8*			2		5	18	23
Sludge 1	2,3,7,8-TCDF	10.033	6.57%	14.4%	1.85	4.05	4	1	16	8	28
Compost 1	2,3,7,8-TCDF	*2.46*		*26.0*			4		15	5	20
Soil 3	2,3,7,8-TCDF	*0.58*		*78.8*			2		7	16	23

Abbreviations: sr Repeatability standard deviation; SR Reproducibility standard deviation; r Repeatability limit (comparing two measurements); R Reproducibility limit (comparing two measurements); p Number of labs; */* determination not possible.

1.4 Annexes

Annex 1: Model questionnaire to be filled by the participating laboratories

Annex 3: Report on the validation materials used

Annex 2: Statistical calculations

Annex 3: Data submitted

Annex 1:

Model questionnaire to be filled by the participating laboratories

Model questionnaire to be filled by the participating laboratories

Name of laboratory:
Contact person:
Contact details: email:
Phone:
Fax:
Mail address of lab:

Dispatch address of lab for shipment of samples (no PO boxes!):

Title of measurement method (copy attached):

Our laboratory is willing to participate in the precision experiment for this draft standard method.

Yes ☐ No ☐

As participant we understand that:

- All essential apparatus, chemicals and other requirements specified in the method must be available in our laboratory when the programme begins
- Specified timing requirements such as starting and finishing date of the programme must be rigidly met
- The method must be strictly adhered to
- Samples must be handled in accordance with instructions
- A qualified operator must perform the measurements

Having studied the method and having made a fair appraisal of our capabilities and facilities, we feel that we will be adequately prepared for cooperative testing of this method.

Comments:

…….
Signature Date

Annex 2:

Report on the validation materials used

Abstract

This report gives an overview on the available analytical information on the following raw materials to be used for the production of validation materials of the so-called Project HORIZONTAL:

- Four sludge materials from Düsseldorf, Germany,
- An agricultural soil material from Reading, United Kingdom;
- A compost material from Vienna, Austria;
- A compost material from Korschenbroich, Germany;
- A sludge-amended, agricultural soil from Pavia Province, Italy;
- A sludge-amended soil from Barcelona, Spain
- A sludge-amended soil from Essen, Germany
- A long-term sludge exposed soil from Hohenheim, Germany

List of Abbreviations

Throughout this report the following abbreviations are used.

AOX	absorbable organic halogens
C_{org}	organic carbon content
C_{total}	total carbon content
CAT	cation exchangeable
CDD	chlorinated dibenzodioxin
CDF	chlorinated dibenzofuran
DEHP	di(2-ethylhexyl)phthalate
DM	dry matter
EPA	Environment Protection Agency
EU	European Union
FM	fresh matter
Hp	hepta
Hx	hexa
IES	Institute for Environment and Sustainability
IRMM	Institute for Reference Materials and Measurements
JRC	Joint Research Centre
LAS	linear alkylsulfonates
LoD	limit of detection
LUA	Landesumweltamt
N_{total}	total nitrogen content
NH_4-N	Ammonium nitrogen
NO_3-N	Nitrate nitrogen
NP	nonylphenol
NRW	North Rhine Westphalia
O	octa
P	poly
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
Pe	penta
T	tetra
TEQ	toxicity equivalent
UBA	Umweltbundesamt
WHO	World Health Organization
WWTP	waste water treatment plant

1 Introduction

This report gives an overview on the available analytical information on the following raw materials to be used for the production of validation materials of the so-called Project HORIZONTAL:

- Four sludge materials from Düsseldorf, Germany,
- An agricultural soil material from Reading, United Kingdom;
- A compost material from Vienna, Austria;
- A compost material from Korschenbroich, Germany;
- A sludge-amended, agricultural soil from Pavia Province, Italy;
- A sludge-amended soil from Barcelona, Spain
- A sludge-amended soil from Essen, Germany
- A long-term sludge exposed soil from Hohenheim, Germany

The following analytical information was gathered partly before and during the sampling of the raw materials, to be used for the production of the HORIZONTAL validation materials. The material were sampled by IES and shipped to IRMM in the course of the year 2005. The information gathered was then completed by various analytical screenings for PAHs and PCBs done by the Institute for Reference Materials and Measurements, Geel, Belgium, for phthalates done by UBA, Berlin, Germany, for PBDE done by IIQAB-CSIC, Barcelona, Spain, for trace elements and some selected major and minor elements by the Institute for Environment and Sustainability, Ispra, Italy.

The work compiled hereafter is based on the numerous additional efforts of the scientists working at various members of the consortium Project HORIZONTAL-Org and contributing organisations.

This work is gratefully acknowledged.

2 Overview on property values

2.1 Sludge materials from Düsseldorf, Germany

The various sewage sludge materials originate from various installations in the North Rhine Westphalia and were produced and sampled by staff from the Landesumweltamt (LUA) NRW under the responsibility from Dr. K. Furtmann.

In total, four sludge materials (Sludge A and D from a major municipal WWTP, Sludge B from a municipal WWTP with industrial input, and Sludge C from a municipal WWTP with high PCB-Content,) were obtained and will be blended to two final materials. Before sampling the following analytical data for a typical sample were received.

Table 1 – Analytical data obtained on an average sludge sample in LUA NRW (with courtesy of K. Furtmann, LUA, Düsseldorf)

Parameter	*Concentration*
PCB	120 ug/kg
DEHP	110 mg/kg
PAH	5 mg/kg (EPA)
PCDD/F	15 ng TE/kg
PBDE	400 ug/kg
NP	40 mg/kg
LAS	3 g/kg
AOX	300 mg/kg

Subsequent screening led to the information displayed hereafter. It should be stressed that the data were obtained as SCREENING information on the UNTREATED or partially treated raw materials. Therefore, the final target values, which are relevant for the validation intercomparison can be different.

Table 2 – Analytical data obtained on a first screening on the sludge samples from LUA NRW

	Sewage sludge A Dusseldorf	*sewage sludge D Dusseldorf*
PCB (ng/g)		
28	62	35
52	101	65
101	31	38
118	49	40
153	30	33
105	24	11
138	46	38
156	<1	<1
180	34	23
170	23	19
PAH (ng/g)		
Naphtalene	34	381
Acenaphtylene	15	43
Acenaphthene	81	108
Fluorene	94	1167
Phenantrene		3440
Anthracene	22	344
Flouranthene	316	4817
Pyrene	235	3011
Benz(a)anthracene	473	791
Chrysene	691	1078
Benz(b)fluoranthene	538	1688
Benz(k)fluoranthene	228	635
Benz(a)pyrene	383	1114
Indeno(1,2,3-c,d)pyrene	92	229
Dibenzo(a,h)anthracene	71	70
Benzo(g,h,i)perylene	80	185

Table 3 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	*DBP*	*DCHP*	*DEHP*	*Water*
	µg/g dm	µg/g dm	µg/g dm	µg/g dm	Wgt. %
Sludge D (1)		0.135		41.474	3.85
Sludge B (2)	0.538	0.034		30.634	5.47
Sludge A (3)	0.184	0.037		31.399	1.46
Sludge C (4)		0.354	1.528	6.678	2.29

Table 4 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Sludge 2 (B)
Tetra-BDE-47	55.4
Penta-BDE-100	9.59
Penta-BDE-99	69.4
Hexa-BDE-154	5.91
Hexa-BDE-153	7.72
Hepta-BDE-183	5.09
Octa-BDE-196	nq
Octa-BDE-197	nq
Octa-BDE-203	9.70
Deca-BDE-209	2216
TOTAL	2379

Table 5 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena, IES, Ispra, Spain). Note that these data are based on single measurements!

	Cd	***Co***	***Cr***	***Cu***	***Mn***	***Ni***	***Pb***	***Sb***	***Tl***	***V***	***Zn***
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Sludge 1 (D)	2.65	29.0	53.3	359	1231	33.8	78.4	4.38	< 0.05	23.2	786
Sludge 2 (B)	1.19	31.1	62.6	202	278	29.9	72.2	2.51	< 0.05	11.8	625
Sludge 3 (A)	1.68	36.0	62.1	332	847	41.6	119	4.51	< 0.05	11.6	1237
Sludge 4 (C)	5.63	19.8	116	273	726	51.1	473	6.18	< 0.05	44.4	2015

Table 6 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	***SiO2 (%)***	***Al2O3 (%)***	***CaO (%)***	***K2O (%)***	***Fe2O3 (%)***	***MgO (%)***	***TiO2 (PPM)***	***S (PPM)***	***P2O5 (PPM)***
Sludge 1 (D)	21.54	5.8	8.44	0.99	10.3	1.01	4367	<15	50448
Sludge 2 (B)	10.67	3.66	6.92	0.46	14.91	0.77	5217	<15	57633
Sludge 3 (A)	7.31	6.63	6.84	0.35	12.87	0.68	3733	<15	60369
Sludge 4 (C)	43.79	9.65	5.27	1.63	5.22	1.07	5628	<15	23945

Sample	***Na2O (%)***	***Cl (PPM)***	***Pb (PPM)***	***Zn (PPM)***	***Cu (PPM)***	***Ni (PPM)***	***Mn (PPM)***	***Cr (PPM)***
Sludge 1 (D)	0.3	2403	101	1002	350	15	1944	132
Sludge 2 (B)	0.31	315	97	879	172	12	514	180
Sludge 3 (A)	0.31	1281	153	1567	265	16	1440	168
Sludge 4 (C)	0.55	231	628	2625	371	81	1101	244

2.2 Agricultural soil material from Reading, United Kingdom

The material was proposed by the University of Reading (S. Nortcliff) and was sampled from a site called "*Frogmore Farm*" which was featured in the "*Metals*" Report for HORIZONTAL. This site is close to Reading with soils developed on flintyloamy periglacial materials over Chalk, has a long and well documented history of sludge application. The focus of the work of Nortcliff *et al.* undertook at this site and the monitoring and control at the site (by Thames Water and the subsequent subsidiary bodies dealing with sludge application to soil) was on metals (and metal loads), with no analysis or indeed any form of investigation in to organics in the broadest sense.

The analytical information produced in the context of the screening of the raw material is displayed below.

Table 7 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	*DBP*	*DCHP*	*DEHP*	*Water*
	µg/g dm	µg/g dm	µg/g dm	µg/g dm	Wgt. %
Soil 3 (Reading)		0.032		0.119	6.69

Table 8 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	***Co***	***Cr***	***Cu***	***Mn***	***Ni***	***Pb***	***Sb***	***Tl***	***V***	***Zn***
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Soil 3 (Reading)	0.15	7.06	27.9	13.8	152	9.01	26.7	3.00	< 0.05	25.8	93.1

Table 9 – Analytical data obtained on a first screening on the sludge-amended soil from Reading (courtesy of IRMM)

Parameter	*Concentration*
PCB	**ng/g**
28	<1
52	<1
101	<1
118	<1
153	<1
105	<1
138	<1
156	<1
180	<1
170	<1
PAH	**ng/g**
Naphtalene	<10
Acenaphtylene	21
Acenaphthene	<10
Fluorene	<10
Phenantrene	<10
Anthracene	<10
Flouranthene	818
Pyrene	776
Benz(a)anthracene	565

Parameter	*Concentration*
Chrysene	608
Benz(b)fluoranthene	824
Benz(k)fluoranthene	329
Benz(a)pyrene	799
Indeno(1,2,3-c,d)pyrene	779
Dibenzo(a,h)anthracene	118
Benzo(g,h,i)perylene	394

Table 10 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Soil 3 (Reading)
Tetra-BDE-47	nq
Penta-BDE-100	nq
Penta-BDE-99	1.03
Hexa-BDE-154	0.03
Hexa-BDE-153	nq
Hepta-BDE-183	nq
Octa-BDE-196	nq
Octa-BDE-197	nd
Octa-BDE-203	nd
Deca-BDE-209	272
TOTAL	273

Table 11 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	***Co***	***Cr***	***Cu***	***Mn***	***Ni***	***Pb***	***Sb***	***Tl***	***V***	***Zn***
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Soil 3 (Reading)	0.15	7.06	27.9	13.8	152	9.01	26.7	3.00	< 0.05	25.8	93.1

Table 12 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	***SiO2 (%)***	***Al2O3 (%)***	***CaO (%)***	***K2O (%)***	***Fe2O3 (%)***	***MgO (%)***	***TiO2 (PPM)***	***S (PPM)***	***P2O5 (PPM)***
Soil 3 (Reading)	79.36	4.77	1.12	0.96	1.94	0.17	4107	443	2102

Sample	***Na2O (%)***	***Cl (PPM)***	***Pb (PPM)***	***Zn (PPM)***	***Cu (PPM)***	***Ni (PPM)***	***Mn (PPM)***	***Cr (PPM)***
Soil 3 (Reading)	0.42	13	45	69	69	69	216	92

Table 13 – Screening data on mercury by solid-sampling cold-vapour AAS using amalgamation enrichment (with courtesy of G. Locoro).

Sample	***Hg*** *µg/g*
Soil 3 (Reading)	0.12

2.3 Compost from Vienna, Austria

The fresh compost material was obtained from the Austrian Federal Environment Agency (UBA, Vienna), which had used a sub-batch of the raw material for national intercomparson. The remainder of the material was stored at 4°C until shipment to IRMM for further processing. The following analytical information was provided by UBA Austria and completed with various screenings.

Table 14 – Analytical data on compost material received from UBA Austria
Inorganic and sum parameters

Parameter	*Unit*	*Sample fraction used*	*Observed mean*
B CAT	mg/l F.M.	Fresh sample, <10mm	6.1
K CAT	mg/l F.M.	Fresh sample, <10mm	2624
Mg CAT	mg/l F.M.	Fresh sample, <10mm	242
P CAT	mg/l F.M.	Fresh sample, <10mm	49
B CAT	% D.M.	Fresh sample, <10mm	0.0017
K CAT	% D.M.	Fresh sample, <10mm	0.72
Mg CAT	% D.M.	Fresh sample, <10mm	0.07
P CAT	% D.M.	Fresh sample, <10mm	0.01
NO3-N	mg/kg F.M.	Fresh sample, <10mm	3.5
NH4-N	mg/kg F.M.	Fresh sample, <10mm	230
Ctotal	% D.M.	<45°dry, milled	29
Corg	% D.M.	<45°dry, milled	27
Ntotal	% D.M.	<45°dry, milled	1.7
P	mg/kg D.M.	<45°dry, milled	2596
K	mg/kg D.M.	<45°dry, milled	11019
K	% D.M.	<45°dry, milled	1.10
B	mg/kg D.M.	<45°dry, milled	60
Cd	mg/kg D.M.	<45°dry, milled	0.46
Cr	mg/kg D.M.	<45°dry, milled	25
Cu	mg/kg D.M.	<45°dry, milled	46
Hg	mg/kg D.M.	<45°dry, milled	0.20
Ni	mg/kg D.M.	<45°dry, milled	18
Pb	mg/kg D.M.	<45°dry, milled	45
Zn	mg/kg D.M.	<45°dry, milled	198
Ca	mg/kg D.M.	<45°dry, milled	68776
Ca	% D.M.	<45°dry, milled	6.9
Mo	mg/kg D.M.	<45°dry, milled	0.8
S	mg/kg D.M.	<45°dry, milled	2137
Fe	mg/kg D.M.	<45°dry, milled	9959
Mn	mg/kg D.M.	<45°dry, milled	418
Na	mg/kg D.M.	<45°dry, milled	742
Co	mg/kg D.M.	<45°dry, milled	4.1
AOX	mg/kg D.M.	<30° dry, milled	62

Table 15 – Analytical data on compost material received from UBA Austria
Polycyclic aromatic hydrocarbons

PAH	*Unit*	*Result*
Naphthaline	µg/kg DM	9.3
Acenaphthylene	µg/kg DM	8.6
Acenaphthene	µg/kg DM	5
Fluorene	µg/kg DM	8.0
Phenanthrene	µg/kg DM	89
Anthracene	µg/kg DM	27
Fluoranthene	µg/kg DM	487
Pyrene	µg/kg DM	380

PAH	*Unit*	*Result*
Benzo(a)anthracene	µg/kg DM	278
Chrysene	µg/kg DM	317
Benzo(b)fluoranthene	µg/kg DM	365
Benzo(k)fluoranthene	µg/kg DM	193
Benz(a)pyrene	µg/kg DM	320
Indeno(1,2,3-c,d)pyrene	µg/kg DM	233
Dibenz(a,h)anthracene	µg/kg DM	67
Benzo(g,h,i)perylene	µg/kg DM	225
Sum EPA	µg/kg DM	3013
Sum EPA	mg/kg DM	3.0

Table 16 – Analytical data on compost material received from UBA Austria
Sum PCDDs and PCBs

Parameter			
Dioxine	TEQ (ITEF)	ng/kg DM	7.3
PCB	TEQ (WHO)	ng/kg DM	3.5
	Σ Ballschmiter	mg/kg DM	0.05

Table 17 – Analytical data on compost material obtained by screening in IRMM

Parameter	*Result in ng/g*
PCB	
28	2
52	2
101	4
118	3
153	10
105	1
138	8
156	1
180	5
170	<1
PAH	
Naphtalene	<10
Acenaphtylene	<10
Acenaphthene	<10
Fluorene	<10
Phenantrene	<10
Anthracene	26
Fluoranthene	611
Pyrene	510
Benz(a)anthracene	888
Chrysene	957
Benz(b)fluoranthene	1531
Benz(k)fluoranthene	547
Benz(a)pyrene	1101
Indeno(1,2,3-c,d)pyrene	416
Dibenzo(a,h)anthracene	81
Benzo(g,h,i)perylene	295

Table 18 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Compost 1 (Vienna)
Tetra-BDE-47	4.02
Penta-BDE-100	0.19
Penta-BDE-99	2.59
Hexa-BDE-154	nq
Hexa-BDE-153	0.23
Hepta-BDE-183	0.04
Octa-BDE-196	nq
Octa-BDE-197	nq
Octa-BDE-203	1.44
Deca-BDE-209	17.4
TOTAL	25.9

Table 19 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	*DBP*	*DCHP*	*DEHP*	*Water*
	µg/g dm	µg/g dm	µg/g dm	µg/g dm	Wgt. %
Compost 1 (Vienna)		0.058		1.426	5.57

Table 20 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	***Co***	***Cr***	***Cu***	***Mn***	***Ni***	***Pb***	***Sb***	***Tl***	***V***	***Zn***
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Compost 1 (Vienna)	0.39	7.36	31.9	41.0	365	12.7	49.5	0.04	0.79	0.13	208

Table 21 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	***SiO2 (%)***	***Al2O3 (%)***	***CaO (%)***	***K2O (%)***	***Fe2O3 (%)***	***MgO (%)***	***TiO2 (PPM)***	***S (PPM)***	***P2O5 (PPM)***
Compost 1 (Vienna)	20.63	4.31	6.17	4.26	1.99	2.49	1602	<15	10521

Sample	***Na2O (%)***	***Cl (PPM)***	***Pb (PPM)***	***Zn (PPM)***	***Cu (PPM)***	***Ni (PPM)***	***Mn (PPM)***	***Cr (PPM)***
Compost 1 (Vienna)	0.35	3496	81	375	79	55	653	60

Table 22 – Screening data on mercury by solid-sampling cold-vapour AAS using amalgamation enrichment (with courtesy of G. Locoro).

Sample	***Hg*** *µg/g*
Compost 1 (Vienna)	0.17

2.4 Agricultural soil, sludge amended soil from Pavia, Italy

This sludge-amended soil material was obtained during a monitoring campaign, which aimed at a generic description of the over-all soil quality in Pavia Province, Italy. The material, which was collected from the upper horizon, originates from a small farm called "*Cascina Novello*". During the characterisation of the site, the following analytical information was obtained on a pooled sample of a sub-area of the farm of 20 X 20 m^2.

Table 23 – Analytical data on Pavia soil

Parameter	*Result*
Al	7.13 Wgt%
As	22.4 mg/kg
Cd	0.79 mg/kg
Cr	59 mg/kg
Cu	30.8 mg/kg
Hg	0.08 mg/kg
Ni	34.4 mg/kg
Pb	24.6 mg/kg
Zn	95 mg/kg
C	0.91 Wgt %
2,3,7,8-TCDD	0.047 pg/g
1,2,3,7,8-PeCDD	0.15 pg/g
1,2,3,4,7,8-HxCDD	0.19 pg/g
1,2,3,6,7,8-HxCDD	1.5 pg/g
1,2,3,7,8,9-HxCDD	0.74 pg/g
1,2,3,4,6,7,8-HpCDD	26 pg/g
OCDD	382 pg/g
2,3,7,8-TCDF	0.68 pg/g
1,2,3,7,8-PeCDF	0.53 pg/g
2,3,4,7,8-PeCDF	0.71 pg/g
1,2,3,4,7,8-HxDF	1.00 pg/g
1,2,3,6,7,8-HxDF	0.66 pg/g
2,3,4,6,7,8-HxDF	1.6 pg/g
1,2,3,7,8,9-HxDF	0.27 pg/g
1,2,3,4,6,7,8-HpDF	12 pg/g
1,2,3,4,7,8,9-HpDF	0.68 pg/g
OCDF	33 pg/g
I-TEQ	2.0 pg/g
WHO-TEQ	1.7 pg/g

In addition, the screening performed at IRMM did not reveal significant quantities of PCBs and PAHs, which were all below the LoDs (1 ng/g for PCBs and 10 ng/g for PAHs, respectively).

Table 24 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	*DBP*	*DCHP*	*DEHP*	*Water*
	µg/g TM	µg/g TM	µg/g TM	µg/g TM	Wgt. %
Soil 5 (Pavia)		0.005		0.011	1.54

Table 25 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Soil 5 (Pavia)
Tetra-BDE-47	nq
Penta-BDE-100	nq
Penta-BDE-99	0.39
Hexa-BDE-154	nq
Hexa-BDE-153	nq
Hepta-BDE-183	0.08
Octa-BDE-196	nq
Octa-BDE-197	nd
Octa-BDE-203	nd
Deca-BDE-209	670
TOTAL	671

Table 26 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	***Co***	***Cr***	***Cu***	***Mn***	***Ni***	***Pb***	***Sb***	***Tl***	***V***	***Zn***
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Soil 5 (Pavia)	0.33	18.4	57.3	22.5	426	30.5	20.6	2.00	< 0.05	38.1	87.8

Table 27 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	***SiO2 (%)***	***Al2O3 (%)***	***CaO (%)***	***K2O (%)***	***Fe2O3 (%)***	***MgO (%)***	***TiO2 (PPM)***	***S (PPM)***	***P2O5 (PPM)***
Soil 5 (Pavia)	69.39	12.9	1.45	2.24	4.25	1.16	6118	255	1789

Sample	***Na2O (%)***	***Cl (PPM)***	***Pb (PPM)***	***Zn (PPM)***	***Cu (PPM)***	***Ni (PPM)***	***Mn (PPM)***	***Cr (PPM)***
Soil 5 (Pavia)	1.84	62	38	108	55	66	597	110

Table 28 – Screening data on mercury by solid-sampling cold-vapour AAS using amalgamation enrichment (with courtesy of G. Locoro).

Sample	***Hg*** *µg/g*
Soil 5 (Pavia)	0.06

2.5 Sludge-amended-soil from Barcelona, Spain

The sludge-amended soil material from Barcelona sampled upon indication from the Barcelo'- Group in Barcelona.

Table 29 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	*DBP*	*DCHP*	*DEHP*	*Water*
	µg/g dm	µg/g dm	µg/g dm	µg/g dm	Wgt. %
Soil 2 (Lleida T.)		0.015		0.183	11.38

Table 30 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Soil 2 (Lleida T.)
Tetra-BDE-47	nq
Penta-BDE-100	nq
Penta-BDE-99	1.59
Hexa-BDE-154	0.45
Hexa-BDE-153	nq
Hepta-BDE-183	0.48
Octa-BDE-196	1.60
Octa-BDE-197	nq
Octa-BDE-203	nq
Deca-BDE-209	1000
TOTAL	1004

Table 31 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	***Co***	***Cr***	***Cu***	***Mn***	***Ni***	***Pb***	***Sb***	***Tl***	***V***	***Zn***
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Soil 2 (Lleida T.)	0.59	14.1	32.7	53.6	405	18.6	18.4	2.24	< 0.05	31.8	111

Table 32 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	***SiO2 (%)***	***Al2O3 (%)***	***CaO (%)***	***K2O (%)***	***Fe2O3 (%)***	***MgO (%)***	***TiO2 (PPM)***	***S (PPM)***	***P2O5 (PPM)***
Soil 2 (Lleida T.)	44.43	10.67	14.29	2.53	3.44	2.04	4116	780	3396

Sample	***Na2O (%)***	***Cl (PPM)***	***Pb (PPM)***	***Zn (PPM)***	***Cu (PPM)***	***Ni (PPM)***	***Mn (PPM)***	***Cr (PPM)***
Soil 2 (Lleida T.)	0.64	65	26	125	59	17	547	65

Table 33 – Screening data on mercury by solid-sampling cold-vapour AAS using amalgamation enrichment (with courtesy of G. Locoro).

Sample	***Hg*** *µg/g*
Soil 2 (Lleida T.)	0.10

2.6 Sludge amended soil from Essen, Germany

The German sludge-amended soil from Essen, which was provided as the three sludge materials by LUA NRW, did not feature significant concentrations of the PCB congeners 28, 52, 101, 118, 153, 105, 138, 156, 180, 170, but had detectable amounts of some PAHs.

Table 34 – Analytical screening data on the German sludge-amended soil.

Parameter	*Concentration (ng/g)*
Naphtalene	<10
Acenaphtylene	<10
Acenaphthene	<10
Fluorene	<10
Phenantrene	<10
Anthracene	<10
Fluoranthene	28
Pyrene	20
Benz(a)anthracene	24
Chrysene	47
Benz(b)fluoranthene	76
Benz(k)fluoranthene	20
Benz(a)pyrene	35
Indeno(1,2,3-c,d)pyrene	35
Dibenzo(a,h)anthracene	10
Benzo(g,h,i)perylene	26

Table 35 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	*DBP*	*DCHP*	*DEHP*	*Water*
	µg/g dm	µg/g dm	µg/g dm	µg/g dm	Wgt. %
Soil 4 (Essen)		0.011		0.302	0.55

Table 36 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Soil 4 (Essen)
Tetra-BDE-47	nq
Penta-BDE-100	nq
Penta-BDE-99	nq
Hexa-BDE-154	nq
Hexa-BDE-153	nq
Hepta-BDE-183	nq
Octa-BDE-196	nq
Octa-BDE-197	nq
Octa-BDE-203	1.28
Deca-BDE-209	19.1
TOTAL	20.3

Table 37 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	***Co***	***Cr***	***Cu***	***Mn***	***Ni***	***Pb***	***Sb***	***Tl***	***V***	***Zn***
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Soil 4 (Essen)	0.52	5.45	26.1	8.05	320	4.03	27.3	2.73	< 0.05	29.5	78.1

Table 38 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	***SiO2 (%)***	***Al2O3 (%)***	***CaO (%)***	***K2O (%)***	***Fe2O3 (%)***	***MgO (%)***	***TiO2 (PPM)***	***S (PPM)***	***P2O5 (PPM)***
Soil 4 (Essen)	79.47	4.42	0.85	0.6	0.86	0.07	2163	189	2019

Sample	***Na2O (%)***	***Cl (PPM)***	***Pb (PPM)***	***Zn (PPM)***	***Cu (PPM)***	***Ni (PPM)***	***Mn (PPM)***	***Cr (PPM)***
Soil 4 (Essen)	0.45	19	42	87	683	60	462	61

Table 39 – Screening data on mercury by solid-sampling cold-vapour AAS using amalgamation enrichment (with courtesy of G. Locoro).

Sample	***Hg*** *µg/g*
Soil 4 (Essen)	0.04

2.7 Long-term sludge exposed soil from Hohenheim-Stuttgart, Germany

Similarly, an additional sludge exposed soil was sampled at the University of Hohenheim, Stuttgart, were a test soil was long-term exposed to elevated concentrations of sewage sludge.

Table 40 – Data on phthalate contents (with courtesy of S. Heise, UBA, Germany)

	DiBP	*DBP*	*DCHP*	*DEHP*	*Water*
	µg/g TM	µg/g TM	µg/g TM	µg/g TM	Wgt. %
Soil 1 (Stuttgart)		0.045		0.263	17.65

Table 41 – Data on PDBE contents (with courtesy of D. Barceló and co-workers, IIQAB-CSIC, Barcelona, Spain)

	Soil 1 (Stuttgart)
Tetra-BDE-47	nq
Penta-BDE-100	nq
Penta-BDE-99	2.30
Hexa-BDE-154	0.06
Hexa-BDE-153	0.04
Hepta-BDE-183	0.04
Octa-BDE-196	nq
Octa-BDE-197	nd
Octa-BDE-203	nd
Deca-BDE-209	498
TOTAL	500

Table 42 – Screening data on some selected trace elements by ICP-AES after micro-wave assisted digestion using aqua regia (with courtesy of F. Sena). Note that these data are based on single measurements!

	Cd	***Co***	***Cr***	***Cu***	***Mn***	***Ni***	***Pb***	***Sb***	***Tl***	***V***	***Zn***
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Soil 1 (Stuttgart)	0.69	12.7	36.1	26.2	504	18.3	25.2	2.62	< 0.05	26.6	142

Table 43 – Screening data on some selected matrix constituents and elements by WDXRF (with courtesy of S. Vaccaro).

Sample	***SiO2 (%)***	***Al2O3 (%)***	***CaO (%)***	***K2O (%)***	***Fe2O3 (%)***	***MgO (%)***	***TiO2 (PPM)***	***S (PPM)***	***P2O5 (PPM)***
Soil 1 (Stuttgart)	71.94	10.06	1.33	1.86	3.66	0.88	7874	275	3571

Sample	***Na2O (%)***	***Cl (PPM)***	***Pb (PPM)***	***Zn (PPM)***	***Cu (PPM)***	***Ni (PPM)***	***Mn (PPM)***	***Cr (PPM)***
Soil 1 (Stuttgart)	1.23	50	47	212	85	69	991	129

Table 44 – Screening data on mercury by solid-sampling cold-vapour AAS using amalgamation enrichment (with courtesy of G. Locoro).

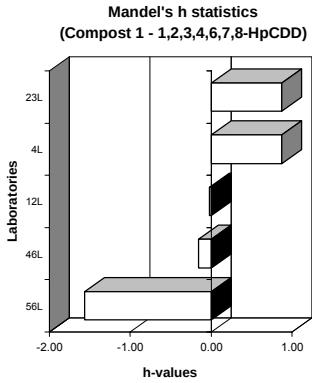
Sample	***Hg*** *µg/g*
Soil 1 (Stuttgart)	1.77

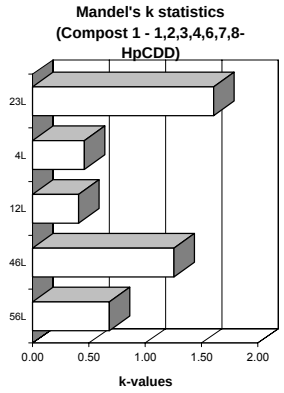
Annex 3:

Statistical calculations

Sample: **Compost 1**

Element: **1,2,3,4,6,7,8-HpCDD**





Unit: ng/kg

Mandel's k statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDD)
Mandel's h statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDD)
Compost 1 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 175.9 [ng/kg]

	General calc.parm.
T1=	3.51713E+03
T2=	6.98259E+05
T3=	20
T4=	80
T5=	2.8576E+03
n=	variabel
p=	5
N-table=	4

Mandel's statistics

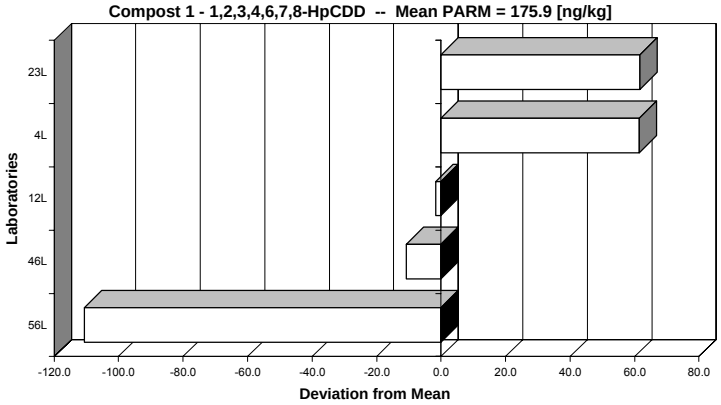
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
56L	65.2000	9.394	4		-1.57	0.68			Fail	65.2000	9.3940		4	3	-110.66
46L	165.0000	17.321	4		-0.15	1.25				165.0000	17.3205		4	3	-10.86
12L	174.3071	5.665	4		-0.02	0.41				174.3071	5.6649		4	3	-1.55
4L	237.2750	6.365	4		0.87	0.46		Fail		237.2750	6.3652		4	3	61.42
23L	237.5000	22.174	4		0.87	1.61	!	Fail		237.5000	22.1736		4	3	61.64
Tot.gem	175.856	12.184 ng/kg		*1%-level:*	1.72	(1.73)			5	*175.8564*	()		5	4	
Tot.std=	70.600	7.251		*5%-level:*	1.57	(1.53)									

End Result:

RESULTS: **Mean =** **175.85643** *ng/kg*

Repeatability variance	**S2r =**	190.50416				
Repeatability std.	**Sr =**	**13.80232**	-->	7.85%	**r =**	38.6465
Between lab variance	**S2L =**	4936.69354				
Reproducibility var.	**S2R =**	5127.19770				
Reproducibility std.	**SR =**	**71.60445**	-->	40.72%	**R =**	200.4925

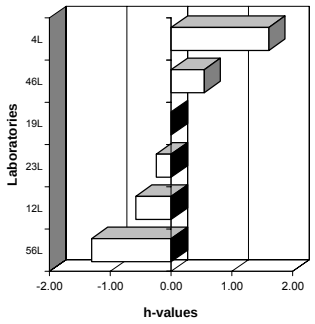
Remarks: **none**



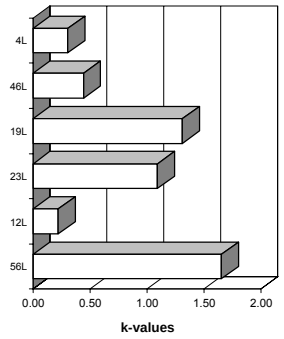
Sample: **Sludge 1**

Element: **1,2,3,4,6,7,8-HpCDD**

Mandel's h statistics
(Sludge 1 - 1,2,3,4,6,7,8-HpCDD)



Mandel's k statistics
(Sludge 1 - 1,2,3,4,6,7,8-HpCDD)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,4,6,7,8-HpCDD)
Mandel's h statistics (Sludge 1 - 1,2,3,4,6,7,8-HpCDD)
Sludge 1 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 185.3 [ng/kg]

	General calc.parm.
T1=	4.44623E+03
T2=	8.44178E+05
T3=	24
T4=	96
T5=	8.3859E+03
n=	variabel
p=	6
N-table=	*4*

Mandel's statistics

End Result:

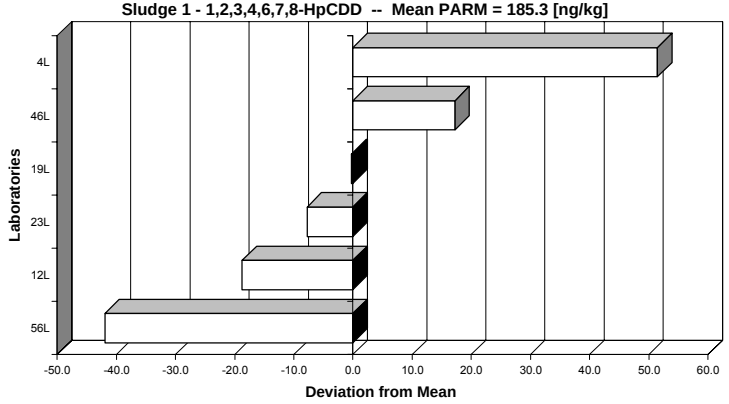
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
56L	143.3000	35.740	4		-1.31	1.66	!		Fail	143.3000	35.7401		4	3	-41.96
12L	166.5336	4.754	4		-0.59	0.22				166.5336	4.7542		4	3	-18.73
23L	177.5000	23.629	4		-0.24	1.09				177.5000	23.6291		4	3	-7.76
19L	185.0000	28.308	4		-0.01	1.31				185.0000	28.3078		4	3	-0.26
46L	202.5000	9.574	4		0.54	0.44				202.5000	9.5743		4	3	17.24
4L	236.7250	6.633	4		1.61	0.31		Fail		236.7250	6.6334		4	3	51.47
Tot.gem	185.260	18.106	ng/kg	*1%-level:*	1.87	(1.77)			6	*185.2598*	()		6	5	
Tot.std=	31.992	12.870		*5%-level:*	1.66	(1.54)									

RESULTS: **Mean =** **185.25977** *ng/kg*

Repeatability variance	**S2r =**	465.88199				
Repeatability std.	**Sr =**	**21.58430**	-->	11.65%	**r =**	60.4360
Between lab variance	**S2L =**	907.02290				
Reproducibility var.	**S2R =**	1372.90489				
Reproducibility std.	**SR =**	**37.05273**	-->	20.00%	**R =**	103.7476

Remarks: **none**

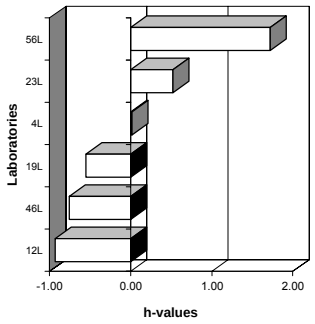
Sludge 1 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 185.3 [ng/kg]



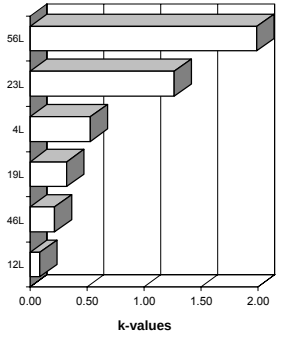
Sample: **Soil 3**

Element: **1,2,3,4,6,7,8-HpCDD**

Mandel's h statistics
(Soil 3 - 1,2,3,4,6,7,8-HpCDD)



Mandel's k statistics
(Soil 3 - 1,2,3,4,6,7,8-HpCDD)



Unit: ng/kg

Mandel's k statistics (Soil 3 - 1,2,3,4,6,7,8-HpCDD)
Mandel's h statistics (Soil 3 - 1,2,3,4,6,7,8-HpCDD)

Soil 3 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 6.54 [ng/kg]

	General calc.parm.
T1=	9.80624E+01
T2=	6.55749E+02
T3=	15
T4=	57
T5=	3.1232E+00
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*		*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*
12L	5.5306	0.137		4		-0.94	0.09			Fail
46L	6.0000	0.346		4		-0.76	0.22			
19L	6.5467	0.519		3		-0.56	0.32			
4L	8.0750	0.850		4		0.01	0.53			
23L	9.4500	2.027		4		0.52	1.27			
56L	12.6750	3.188		4	!	1.72	1.99	!!	Fail	
Tot.gem	8.046	1.178	ng/kg		*1%-level:*	1.87	(1.77)			4
Tot.std=	2.689	1.190			*5%-level:*	1.66	(1.54)			2

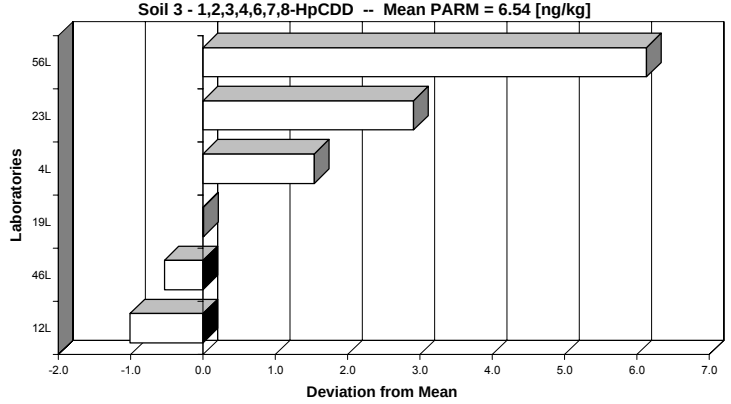
End Result:

PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
5.5306	0.1371		4	3	-1.01
6.0000	0.3464		4	3	-0.54
6.5467	0.5193		3	2	0.01
8.0750	0.8500		4	3	1.54
-	-	,23L	-	-	2.91
-	-	,56L	-	-	6.14
6.5381		(23L ,56L)	4	3	

RESULTS: **Mean =** **6.53806** *ng/kg*

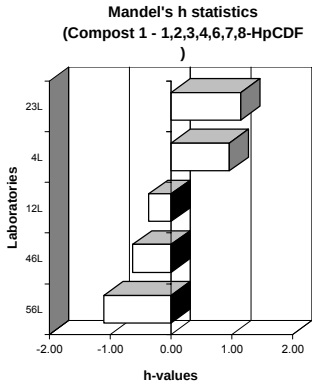
Repeatability variance	**S2r =**	0.28392				
Repeatability std.	**Sr =**	**0.53284**	-->	8.15%	**r =**	1.4920
Between lab variance	**S2L =**	1.23350				
Reproducibility var.	**S2R =**	1.51742				
Reproducibility std.	**SR =**	**1.23184**	-->	18.84%	**R =**	3.4491
Remarks:	**2 Labs rejected! (23L ,56L)**					

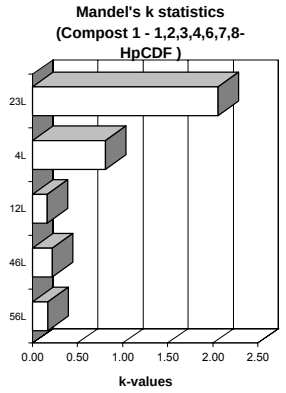
Soil 3 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 6.54 [ng/kg]



Sample: **Compost 1**

Element: **1,2,3,4,6,7,8-HpCDF**





Unit: ng/kg

Mandel's k statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDF)
Mandel's h statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDF)
#NUM!

General calc.parm.

T1=	#NUM!
T2=	#NUM!
T3=	
T4=	
T5=	
n=	variabel
p=	
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
56L	8.3871	1.009	4		-1.11	0.18		
46L	11.5000	1.291	4		-0.63	0.23		
12L	13.2804	0.944	4		-0.36	0.16		
4L	22.0000	4.651	4		0.96	0.81		
23L	23.2500	11.786	4		1.15	2.06	!!	
Tot.gem	15.684	3.936	ng/kg		*1%-level:*	1.72	(1.73)	
Tot.std=	6.589	4.654			*5%-level:*	1.57	(1.53)	

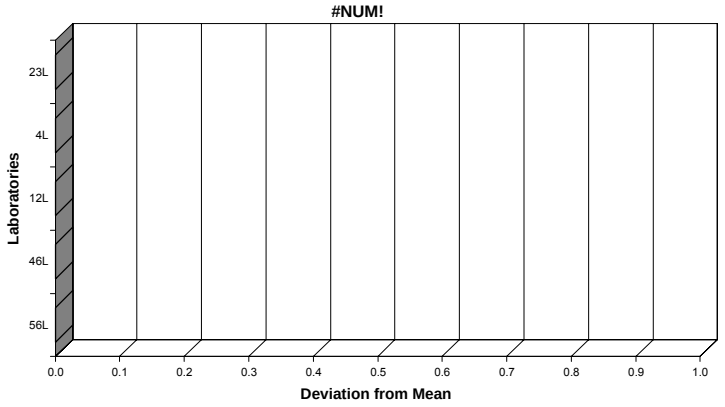
End Result:

PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
#NUM!	- ,56L	-	-		#NUM!
-	- ,46L	-	-		#NUM!
-	- ,12L	-	-		#NUM!
-	- ,4L	-	-		#NUM!
-	- ,23L	-	-		#NUM!
#NUM!	(4L,23L,12L,56L,46L)			-1	

RESULTS: **Mean =** **#NUM!** *ng/kg*

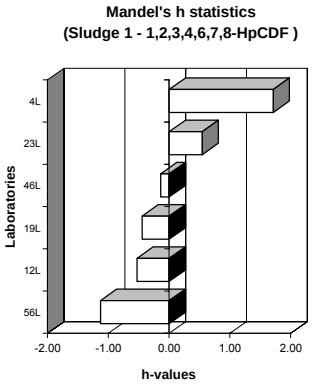
Repeatability variance	**S2r =**	#DIV/0!				
Repeatability std.	**Sr =**	**#DIV/0!**	-->	#DIV/0!	**r =**	#DIV/0!
Between lab variance	**S2L =**	#NUM!				
Reproducibility var.	**S2R =**	#NUM!				
Reproducibility std.	**SR =**	**#NUM!**	-->	#NUM!	**R =**	#NUM!

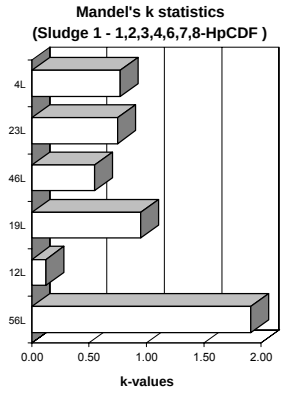
Remarks: **5 Labs rejected! (4L,23L,12L,56L,46L)**



Sample: **Sludge 1**

Element: **1,2,3,4,6,7,8-HpCDF**





Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,4,6,7,8-HpCDF)
Mandel's h statistics (Sludge 1 - 1,2,3,4,6,7,8-HpCDF)
Sludge 1 - 1,2,3,4,6,7,8-HpCDF -- Mean PARM = 95.41 [ng/kg]

General calc.parm.	
T1=	2.28987E+03
T2=	2.24067E+05
T3=	24
T4=	96
T5=	1.3184E+03
n=	variabel
p=	6
N-table=	*4*

Mandel's statistics

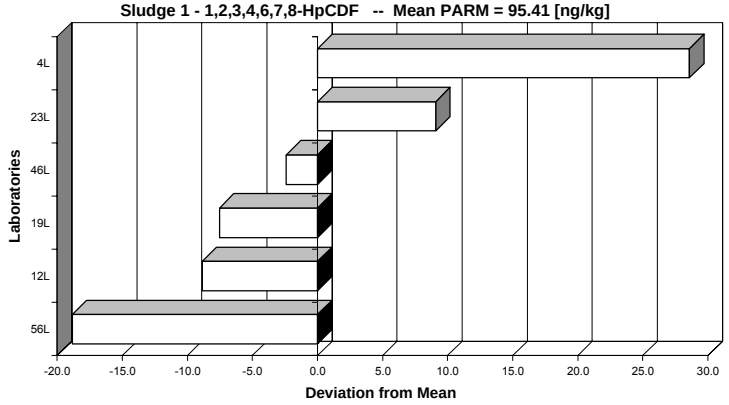
End Result:

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
56L	76.5573	16.319	4		-1.13	1.91	!!		Fail	76.5573	16.3190		4	3	-18.85
12L	86.5354	1.072	4		-0.53	0.13				86.5354	1.0724		4	3	-8.88
19L	87.8750	8.107	4		-0.45	0.95				87.8750	8.1074		4	3	-7.54
46L	93.0000	4.690	4		-0.14	0.55				93.0000	4.6904		4	3	-2.41
23L	104.5000	6.403	4		0.54	0.75				104.5000	6.4031		4	3	9.09
4L	124.0000	6.579	4	!	1.71	0.77		Fail		124.0000	6.5793		4	3	28.59
Tot.gem	95.411	7.195	ng/kg		*1%-level:*	1.87	(1.77)			6	*95.4113*	()	6	5	
Tot.std=	16.714	5.076			*5%-level:*	1.66	(1.54)								

RESULTS: **Mean =** **95.41127** *ng/kg*

Repeatability variance	**S2r =**	73.24592				
Repeatability std.	**Sr =**	**8.55838**	-->	8.97%	**r =**	23.9635
Between lab variance	**S2L =**	261.04540				
Reproducibility var.	**S2R =**	334.29132				
Reproducibility std.	**SR =**	**18.28364**	-->	19.16%	**R =**	51.1942

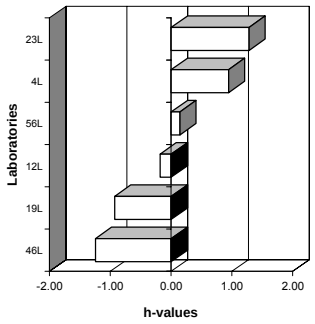
Remarks: **none**



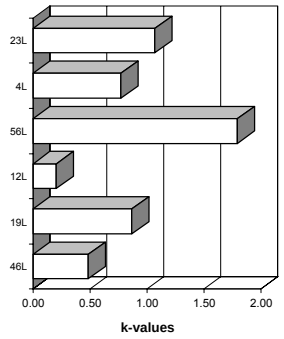
Sample: **Soil 3**

Element: **1,2,3,4,6,7,8-HpCDF**

Mandel's h statistics
(Soil 3 - 1,2,3,4,6,7,8-HpCDF)



Mandel's k statistics
(Soil 3 - 1,2,3,4,6,7,8-HpCDF)



Unit: ng/kg

Mandel's k statistics (Soil 3 - 1,2,3,4,6,7,8-HpCDF)
Mandel's h statistics (Soil 3 - 1,2,3,4,6,7,8-HpCDF)
Soil 3 - 1,2,3,4,6,7,8-HpCDF -- Mean PARM = 12.42 [ng/kg]

General calc.parm.

T1=	2.87261E+02
T2=	3.63801E+03
T3=	23
T4=	89
T5=	1.0541E+02
n=	variabel
p=	6
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
46L	10.4000	1.200	4		-1.25	0.49		
19L	10.9133	2.147	3		-0.93	0.87		
12L	12.1329	0.510	4		-0.18	0.21		
56L	12.6475	4.441	4		0.14	1.80	!!	
4L	13.9500	1.907	4		0.94	0.77		
23L	14.5000	2.646	4		1.28	1.07		
Tot.gem	12.424	2.142 ng/kg		*1%-level:*	1.87	(1.77)		6
Tot.std=	1.622	1.353		*5%-level:*	1.66	(1.54)		

End Result:

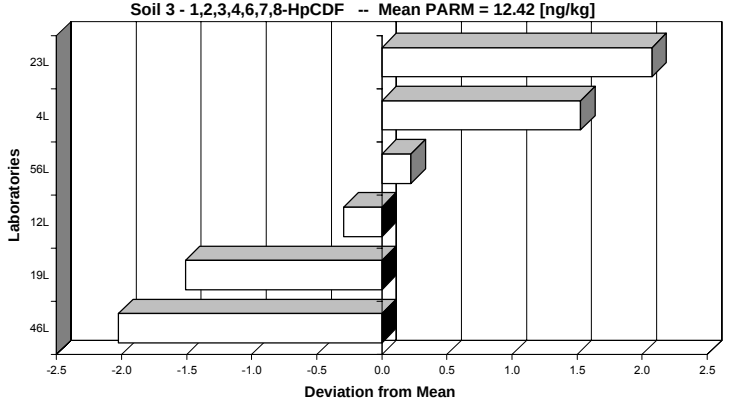
PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
10.4000	1.2000		4	3	-2.02
10.9133	2.1472		3	2	-1.51
12.1329	0.5098		4	3	-0.29
12.6475	4.4413		4	3	0.22
13.9500	1.9070		4	3	1.53
14.5000	2.6458		4	3	2.08
12.4239	()		6	5	

RESULTS: **Mean =** **12.42394** *ng/kg*

Repeatability variance	**S2r =**	6.20036				
Repeatability std.	**Sr =**	**2.49005**	-->	20.04%	**r =**	6.9721
Between lab variance	**S2L =**	1.00491				
Reproducibility var.	**S2R =**	7.20527				
Reproducibility std.	**SR =**	**2.68426**	-->	21.61%	**R =**	7.5159

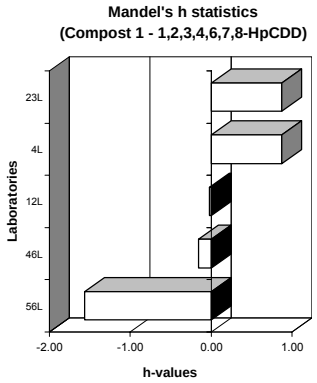
Remarks: **none**

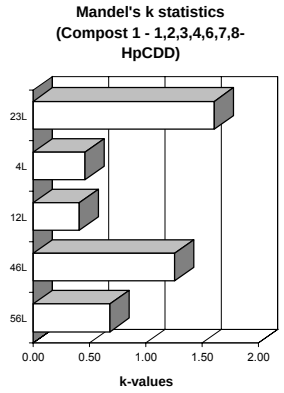
Soil 3 - 1,2,3,4,6,7,8-HpCDF -- Mean PARM = 12.42 [ng/kg]



Sample: **Compost 1**

Element: **1,2,3,4,6,7,8-HpCDD**





Unit: ng/kg

Mandel's k statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDD)
Mandel's h statistics (Compost 1 - 1,2,3,4,6,7,8-HpCDD)
Compost 1 - 1,2,3,4,6,7,8-HpCDD -- Mean PARM = 175.9 [ng/kg]

	General calc.parm.
T1=	3.51713E+03
T2=	6.98259E+05
T3=	20
T4=	80
T5=	2.8576E+03
n=	variabel
p=	*5*
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*
56L	65.2000	9.394	4		-1.57	0.68			Fail
46L	165.0000	17.321	4		-0.15	1.25			
12L	174.3071	5.665	4		-0.02	0.41			
4L	237.2750	6.365	4		0.87	0.46		Fail	
23L	237.5000	22.174	4		0.87	1.61	!	Fail	
Tot.gem	175.856	12.184 ng/kg		*1%-level:*	1.72	(1.73)			5
Tot.std=	70.600	7.251		*5%-level:*	1.57	(1.53)			

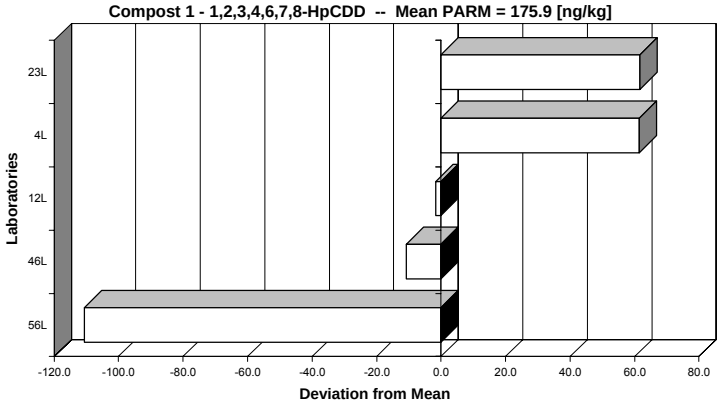
End Result:

PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
65.2000	9.3940		4	3	-110.66
165.0000	17.3205		4	3	-10.86
174.3071	5.6649		4	3	-1.55
237.2750	6.3652		4	3	61.42
237.5000	22.1736		4	3	61.64
175.8564	()		5	4	

RESULTS: **Mean =** **175.85643** *ng/kg*

Repeatability variance	**S2r =**	190.50416				
Repeatability std.	**Sr =**	**13.80232**	-->	7.85%	**r =**	38.6465
Between lab variance	**S2L =**	4936.69354				
Reproducibility var.	**S2R =**	5127.19770				
Reproducibility std.	**SR =**	**71.60445**	-->	40.72%	**R =**	200.4925

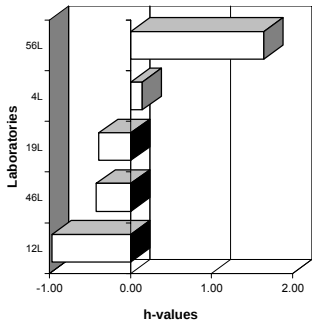
Remarks: **none**



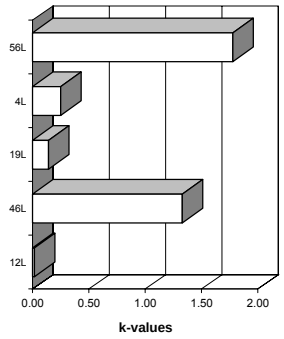
Sample: **Sludge 1**

Element: **1,2,3,4,7,8,9-HpCDF**

Mandel's h statistics
(Sludge 1 - 1,2,3,4,7,8,9-HpCDF)



Mandel's k statistics
(Sludge 1 - 1,2,3,4,7,8,9-HpCDF)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,4,7,8,9-HpCDF)
Mandel's h statistics (Sludge 1 - 1,2,3,4,7,8,9-HpCDF)
Sludge 1 - 1,2,3,4,7,8,9-HpCDF -- Mean PARM = 7.41 [ng/kg]

General calc.parm.	
T1=	1.48141E+02
T2=	1.16636E+03
T3=	20
T4=	80
T5=	4.5943E+01
n=	variabel
p=	5
N-table=	4

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*
12L	5.3948	0.033	4		-0.97	0.02			
46L	6.5250	2.327	4		-0.42	1.33			
19L	6.5900	0.250	4		-0.39	0.14			
4L	7.7000	0.440	4		0.14	0.25			
56L	10.8255	3.105	4	!	1.65	1.77	!!	Fail	
Tot.gem	7.407	1.231	ng/kg		*1%-level:*	1.72	(1.73)		5
Tot.std=	2.078	1.391			*5%-level:*	1.57	(1.53)		

End Result:

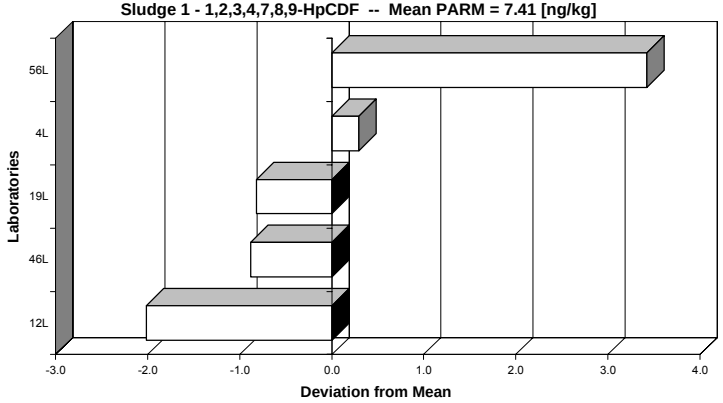
PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
5.3948	0.0328		4	3	-2.01
6.5250	2.3272		4	3	-0.88
6.5900	0.2505		4	3	-0.82
7.7000	0.4397		4	3	0.29
10.8255	3.1051		4	3	3.42
7.4071		()	5	4	

RESULTS: **Mean =** **7.40706** *ng/kg*

Repeatability variance	**S2r =**	3.06290				
Repeatability std.	**Sr =**	**1.75011**	-->	23.63%	**r =**	4.9003
Between lab variance	**S2L =**	3.55091				
Reproducibility var.	**S2R =**	6.61380				
Reproducibility std.	**SR =**	**2.57173**	-->	34.72%	**R =**	7.2008

Remarks: **none**

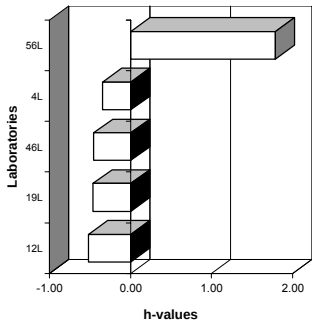
Sludge 1 - 1,2,3,4,7,8,9-HpCDF -- Mean PARM = 7.41 [ng/kg]



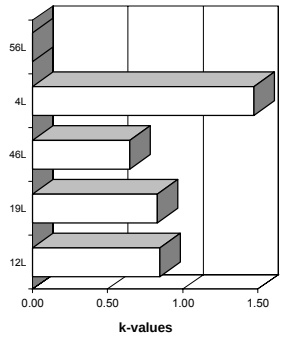
Sample: **Sludge 1**

Element: **1,2,3,4,7,8-HxCDD**

Mandel's h statistics
(Sludge 1 - 1,2,3,4,7,8-HxCDD)



Mandel's k statistics
(Sludge 1 - 1,2,3,4,7,8-HxCDD)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,4,7,8-HxCDD)
Mandel's h statistics (Sludge 1 - 1,2,3,4,7,8-HxCDD)
Sludge 1 - 1,2,3,4,7,8-HxCDD -- Mean PARM = 3.5 [ng/kg]

	General calc.parm.
T1=	5.25284E+01
T2=	1.86187E+02
T3=	15
T4=	57
T5=	6.4029E-01
n=	variabel
p=	4
N-table=	*3*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*
12L	3.0721	0.199	4		-0.52	0.85	
19L	3.3850	0.195	4		-0.47	0.83	
46L	3.4333	0.153	3		-0.46	0.65	
4L	4.1000	0.346	4		-0.34	1.47	
56L	16.5000	-	1	!!	1.79		Fail
Tot.gem	6.098	0.223 ng/kg		*1%-level:*	1.72	(1.77)	
Tot.std=	5.827	0.085		*5%-level:*	1.57	(1.59)	

End Result:

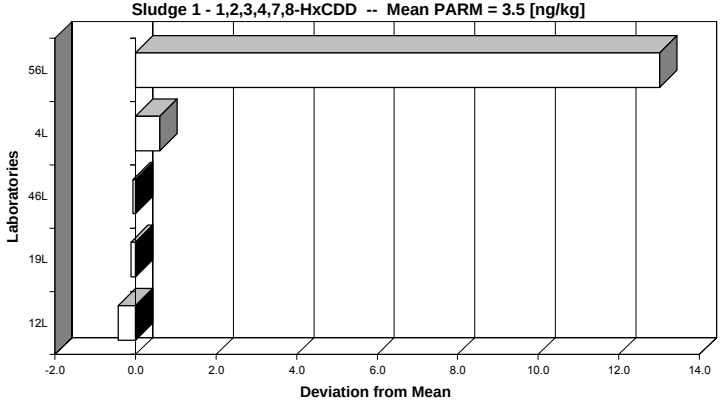
\vX > AvST+2std	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
Fail		3.0721	0.1993		4	3	-0.43
Fail		3.3850	0.1954		4	3	-0.11
Fail		3.4333	0.1528		3	2	-0.06
Fail		4.1000	0.3464		4	3	0.60
	-	-	,56L	-	-		13.00
	4	*3.4976*	(56L)		4	3	
	1						

RESULTS: **Mean =** **3.49761** *ng/kg*

Repeatability variance	**S2r =**	0.05821				
Repeatability std.	**Sr =**	**0.24126**	-->	6.90%	**r =**	0.6755
Between lab variance	**S2L =**	0.18428				
Reproducibility var.	**S2R =**	0.24249				
Reproducibility std.	**SR =**	**0.49243**	-->	14.08%	**R =**	1.3788

Remarks: **1 Lab rejected! (56L)**

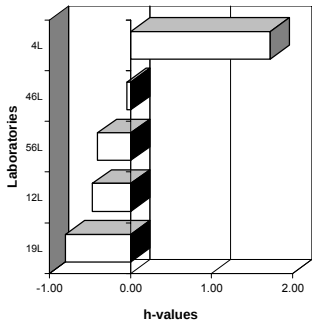
Sludge 1 - 1,2,3,4,7,8-HxCDD -- Mean PARM = 3.5 [ng/kg]



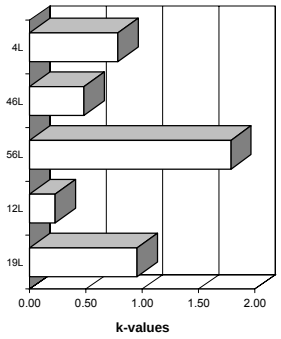
Sample: **Sludge 1**

Element: **1,2,3,6,7,8-HxCDD**

Mandel's h statistics
(Sludge 1 - 1,2,3,6,7,8-HxCDD)



Mandel's k statistics
(Sludge 1 - 1,2,3,6,7,8-HxCDD)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,6,7,8-HxCDD)
Mandel's h statistics (Sludge 1 - 1,2,3,6,7,8-HxCDD)
Sludge 1 - 1,2,3,6,7,8-HxCDD -- Mean PARM = 8.28 [ng/kg]

	General calc.parm.
T1=	1.65567E+02
T2=	1.41503E+03
T3=	20
T4=	80
T5=	1.9356E+01
n=	variabel
p=	5
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
19L	6.9450	1.087	4		-0.80	0.96		
12L	7.4968	0.260	4		-0.47	0.23		
56L	7.6000	2.028	4		-0.41	1.79	!!	
46L	8.2000	0.548	4		-0.05	0.48		
4L	11.1500	0.889	4	!!	1.72	0.78		Fail
Tot.gem	8.278	0.962 ng/kg		*1%-level:*	1.72	(1.73)		
Tot.std=	1.666	0.675		*5%-level:*	1.57	(1.53)		

End Result:

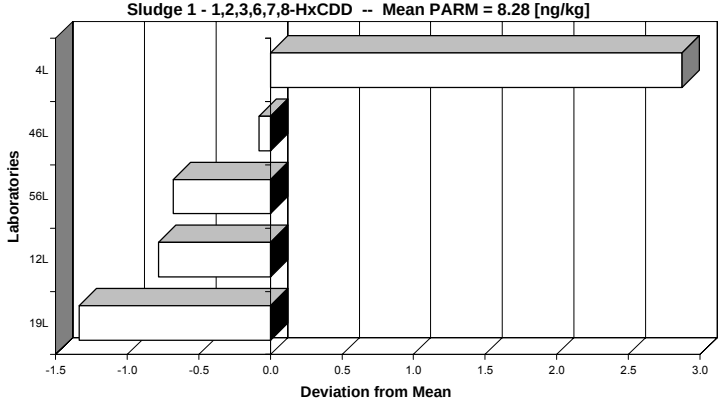
	PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
	6.9450	1.0868		4	3	-1.33
	7.4968	0.2596		4	3	-0.78
	7.6000	2.0281		4	3	-0.68
	8.2000	0.5477		4	3	-0.08
	11.1500	0.8888		4	3	2.87
5	*8.2784*	()		5	4	

RESULTS: **Mean =** **8.27836** *ng/kg*

Repeatability variance	**S2r =**	1.29037				
Repeatability std.	**Sr =**	**1.13594**	-->	13.72%	**r =**	3.1806
Between lab variance	**S2L =**	2.45273				
Reproducibility var.	**S2R =**	3.74310				
Reproducibility std.	**SR =**	**1.93471**	-->	23.37%	**R =**	5.4172

Remarks: **none**

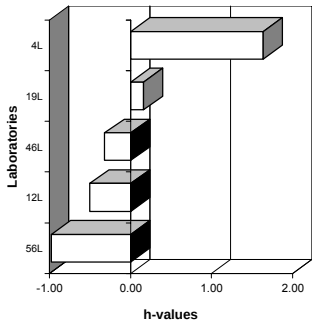
Sludge 1 - 1,2,3,6,7,8-HxCDD -- Mean PARM = 8.28 [ng/kg]



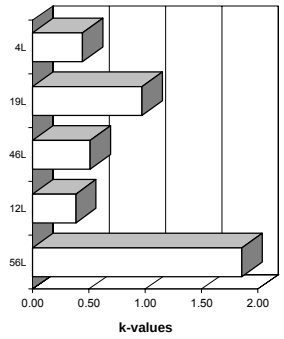
Sample: **Sludge 1**

Element: **1,2,3,6,7,8-HxCDF**

Mandel's h statistics
(Sludge 1 - 1,2,3,6,7,8-HxCDF)



Mandel's k statistics
(Sludge 1 - 1,2,3,6,7,8-HxCDF)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,6,7,8-HxCDF)
Mandel's h statistics (Sludge 1 - 1,2,3,6,7,8-HxCDF)
Sludge 1 - 1,2,3,6,7,8-HxCDF -- Mean PARM = 7.4 [ng/kg]

General calc.parm.	
T1=	1.47943E+02
T2=	1.12613E+03
T3=	20
T4=	80
T5=	9.6995E+00
n=	variabel
p=	5
N-table=	4

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*
56L	6.0250	1.493	4		-0.97	1.86	!!		Fail
12L	6.6857	0.309	4		-0.50	0.38			
46L	6.9500	0.412	4		-0.32	0.51			
19L	7.6250	0.782	4		0.16	0.97			
4L	9.7000	0.356	4	!	1.63	0.44			Fail
Tot.gem	7.397	0.671 ng/kg		*1%-level:*	1.72	(1.73)			
Tot.std=	1.409	0.496		*5%-level:*	1.57	(1.53)			

End Result:

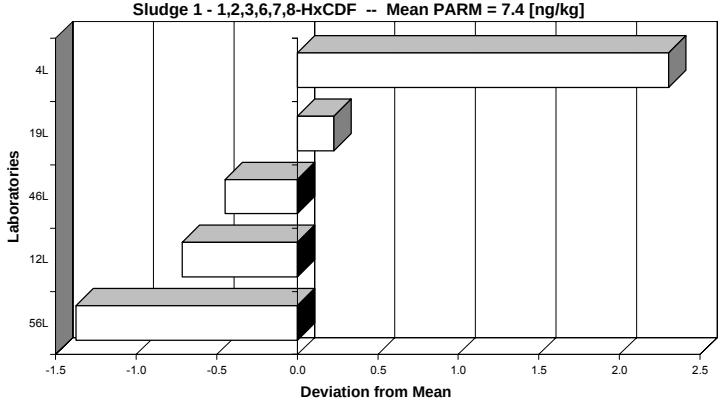
PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
6.0250	1.4930		4	3	-1.37
6.6857	0.3094		4	3	-0.71
6.9500	0.4123		4	3	-0.45
7.6250	0.7820		4	3	0.23
9.7000	0.3559		4	3	2.30
5	*7.3971*	()	5	4	

RESULTS: **Mean =** **7.39714** *ng/kg*

Repeatability variance	**S2r =**	0.64663				
Repeatability std.	**Sr =**	**0.80413**	-->	10.87%	**r =**	2.2516
Between lab variance	**S2L =**	1.82432				
Reproducibility var.	**S2R =**	2.47095				
Reproducibility std.	**SR =**	**1.57193**	-->	21.25%	**R =**	4.4014

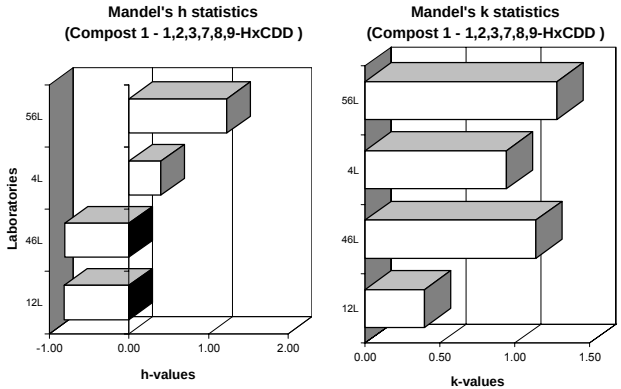
Remarks: **none**

Sludge 1 - 1,2,3,6,7,8-HxCDF -- Mean PARM = 7.4 [ng/kg]



Sample: **Compost 1**

Element: **1,2,3,7,8,9-HxCDD**



Unit: ng/kg

Mandel's k statistics (Compost 1 - 1,2,3,7,8,9-HxCDD)
Mandel's h statistics (Compost 1 - 1,2,3,7,8,9-HxCDD)
Compost 1 - 1,2,3,7,8,9-HxCDD -- Mean PARM = 2.09 [ng/kg]

	General calc.parm.
T1=	2.75803E+01
T2=	5.80691E+01
T3=	14
T4=	52
T5=	4.2296E-01
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
12L	1.5451	0.087	4		-0.82	0.40		Fail
46L	1.5500	0.252	4		-0.81	1.14		Fail
4L	2.3500	0.208	4		0.40	0.94	Fail	
56L	2.9000	0.283	2		1.23	1.28	Fail	
Tot.gem	2.086	0.208	ng/kg	*1%-level:*	1.49	(1.67)		4
Tot.std=	0.661	0.086		*5%-level:*	1.42	(1.5)		

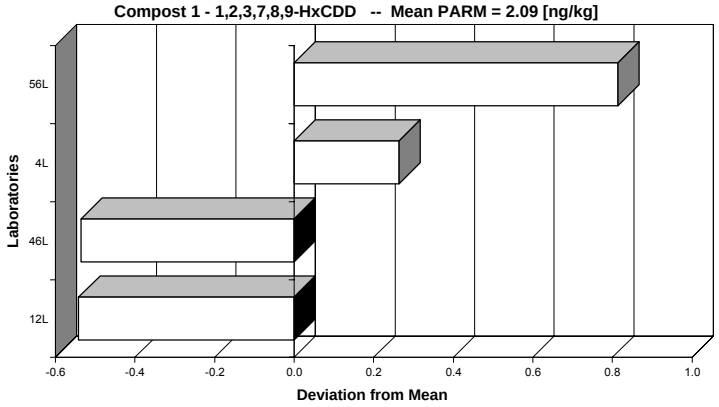
End Result:

PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
1.5451	0.0875		4	3	-0.54
1.5500	0.2517		4	3	-0.54
2.3500	0.2082		4	3	0.26
2.9000	0.2828		2	1	0.81
2.0863	()		4	3	

RESULTS: **Mean =** **2.08627** *ng/kg*

Repeatability variance	**S2r =**	0.04230				
Repeatability std.	**Sr =**	**0.20566**	-->	9.86%	**r =**	0.5758
Between lab variance	**S2L =**	0.35081				
Reproducibility var.	**S2R =**	0.39311				
Reproducibility std.	**SR =**	**0.62698**	-->	30.05%	**R =**	1.7555

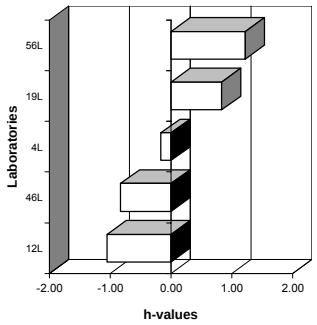
Remarks: **none**



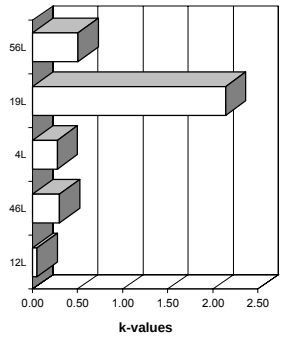
Sample: **Sludge 1**

Element: **1,2,3,7,8,9-HxCDD**

Mandel's h statistics
(Sludge 1 - 1,2,3,7,8,9-HxCDD)



Mandel's k statistics
(Sludge 1 - 1,2,3,7,8,9-HxCDD)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,7,8,9-HxCDD)
Mandel's h statistics (Sludge 1 - 1,2,3,7,8,9-HxCDD)
Sludge 1 - 1,2,3,7,8,9-HxCDD -- Mean PARM = 6.43 [ng/kg]

General calc.parm.	
T1=	1.02832E+02
T2=	7.70893E+02
T3=	16
T4=	64
T5=	1.0314E+01
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
12L	3.9329	0.149	4		-1.05	0.05		
46L	4.5750	0.862	4		-0.83	0.30		
4L	6.5500	0.785	4		-0.17	0.28		
19L	9.5125	6.083	4		0.83	2.14	!!	
56L	10.6500	1.434	4		1.22	0.50		
Tot.gem	7.044	1.863 ng/kg		*1%-level:*	1.72	(1.73)		4
Tot.std=	2.963	2.403		*5%-level:*	1.57	(1.53)		1

End Result:

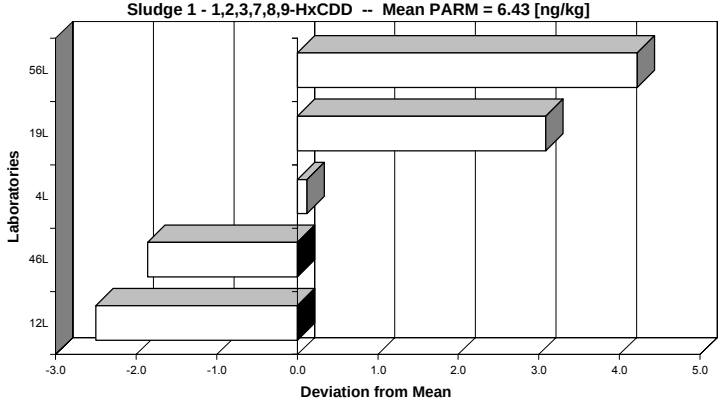
PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
3.9329	0.1493		4	3	-2.49
4.5750	0.8617		4	3	-1.85
6.5500	0.7853		4	3	0.12
-	-	,19L	-	-	3.09
10.6500	1.4341		4	3	4.22
6.4270		(19L)	4	3	

RESULTS: **Mean =** **6.42698** *ng/kg*

Repeatability variance	**S2r =**	0.85953				
Repeatability std.	**Sr =**	**0.92711**	-->	14.43%	**r =**	2.5959
Between lab variance	**S2L =**	8.95154				
Reproducibility var.	**S2R =**	9.81107				
Reproducibility std.	**SR =**	**3.13226**	-->	48.74%	**R =**	8.7703

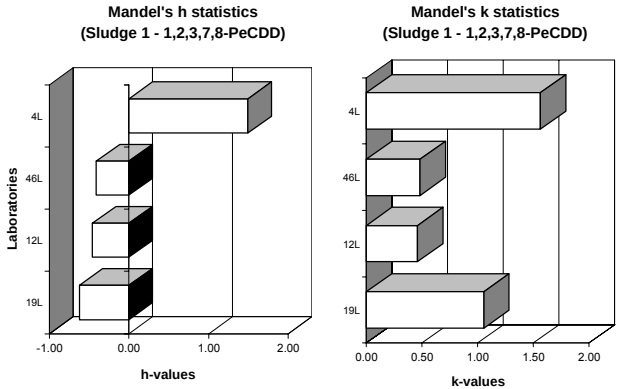
Remarks: **1 Lab rejected! (19L)**

Sludge 1 - 1,2,3,7,8,9-HxCDD -- Mean PARM = 6.43 [ng/kg]



Sample: **Sludge 1**

Element: **1,2,3,7,8-PeCDD**



Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,7,8-PeCDD)
Mandel's h statistics (Sludge 1 - 1,2,3,7,8-PeCDD)
Sludge 1 - 1,2,3,7,8-PeCDD -- Mean PARM = 2.29 [ng/kg]

	General calc.parm.
T1=	3.65631E+01
T2=	1.00307E+02
T3=	16
T4=	64
T5=	2.0500E+00
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*
19L	1.5463	0.436	4		-0.63	1.06			Fail
12L	1.7445	0.190	4		-0.46	0.46			Fail
46L	1.8000	0.200	4		-0.41	0.48			Fail
4L	4.0500	0.645	4	!!	1.49	1.56	!	Fail	
Tot.gem	2.285	0.368	ng/kg		*1%-level:*	1.49	(1.67)		
Tot.std=	1.182	0.217			*5%-level:*	1.42	(1.5)		

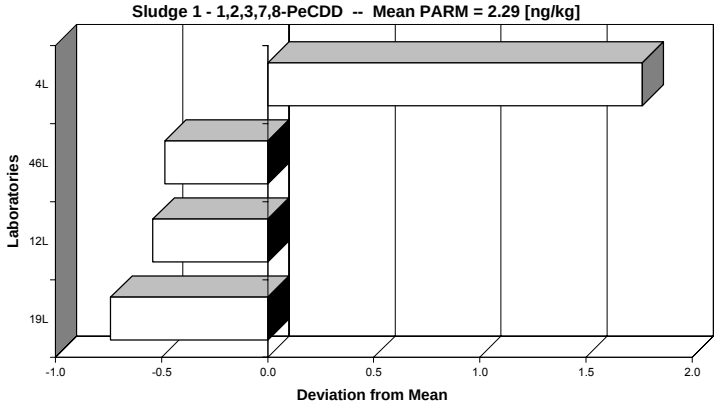
End Result:

	PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
	1.5463	0.4365		4	3	-0.74
	1.7445	0.1901		4	3	-0.54
	1.8000	0.2000		4	3	-0.49
	4.0500	0.6455		4	3	1.76
4	*2.2852*	()		4	3	

RESULTS: **Mean =** **2.28519** *ng/kg*

Repeatability variance	**S2r =**	0.17083				
Repeatability std.	**Sr =**	**0.41332**	-->	18.09%	**r =**	1.1573
Between lab variance	**S2L =**	1.35340				
Reproducibility var.	**S2R =**	1.52423				
Reproducibility std.	**SR =**	**1.23460**	-->	54.03%	**R =**	3.4569

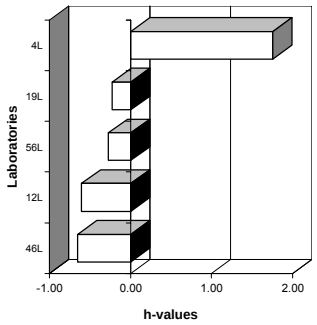
Remarks: **none**



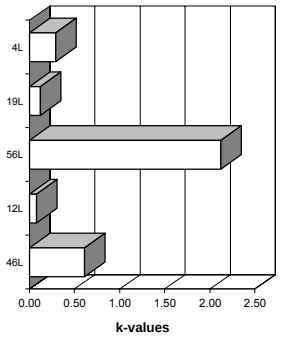
Sample: **Sludge 1**

Element: **1,2,3,7,8-PeCDF**

Mandel's h statistics
(Sludge 1 - 1,2,3,7,8-PeCDF)



Mandel's k statistics
(Sludge 1 - 1,2,3,7,8-PeCDF)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - 1,2,3,7,8-PeCDF)
Mandel's h statistics (Sludge 1 - 1,2,3,7,8-PeCDF)
Sludge 1 - 1,2,3,7,8-PeCDF -- Mean PARM = 5.2 [ng/kg]

	General calc.parm.
T1=	7.87769E+01
T2=	4.29418E+02
T3=	15
T4=	57
T5=	2.0598E+00
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
46L	4.4667	0.850	3		-0.65	0.62		
12L	4.5167	0.114	4		-0.60	0.08		
56L	4.8500	2.937	4		-0.28	2.12	!!	
19L	4.9025	0.170	4		-0.22	0.12		
4L	6.9250	0.403	4	!!	1.76	0.29		
Tot.gem	5.132	0.895 ng/kg		*1%-level:*	1.72	(1.73)		4
Tot.std=	1.021	1.178		*5%-level:*	1.57	(1.53)		

End Result:

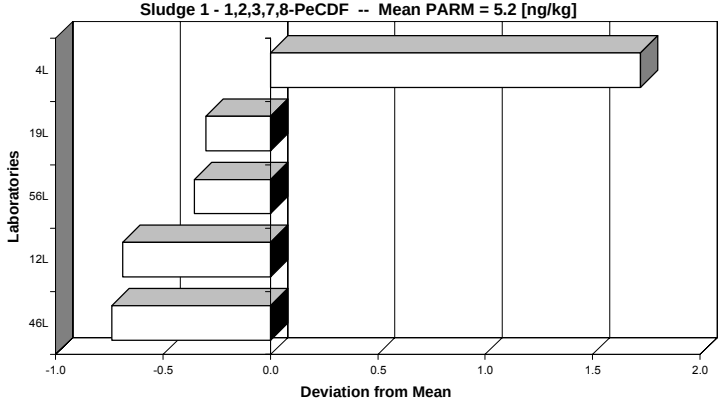
PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
4.4667	0.8505		3	2	-0.74
4.5167	0.1142		4	3	-0.69
-	-	,56L	-	-	-0.35
4.9025	0.1698		4	3	-0.30
6.9250	0.4031		4	3	1.72
5.2027		(56L)	4	3	

RESULTS: **Mean =** **5.20273** *ng/kg*

Repeatability variance	**S2r =**	0.18725				
Repeatability std.	**Sr =**	**0.43272**	-->	8.32%	**r =**	1.2116
Between lab variance	**S2L =**	1.35136				
Reproducibility var.	**S2R =**	1.53861				
Reproducibility std.	**SR =**	**1.24041**	-->	23.84%	**R =**	3.4731

Remarks: **1 Lab rejected! (56L)**

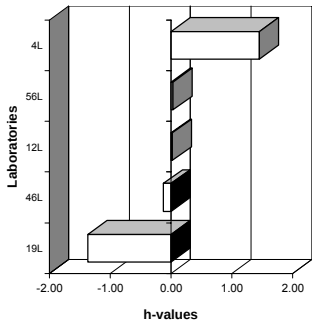
Sludge 1 - 1,2,3,7,8-PeCDF -- Mean PARM = 5.2 [ng/kg]



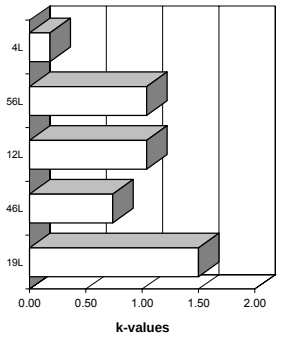
Sample: **Sludge 1**

Element: **2,3,4,7,8-PeCDF**

Mandel's h statistics
(Sludge 1 - 2,3,4,7,8-PeCDF)



Mandel's k statistics
(Sludge 1 - 2,3,4,7,8-PeCDF)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - 2,3,4,7,8-PeCDF)
Mandel's h statistics (Sludge 1 - 2,3,4,7,8-PeCDF)
Sludge 1 - 2,3,4,7,8-PeCDF -- Mean PARM = 9.78 [ng/kg]

General calc.parm.	
T1=	1.95673E+02
T2=	1.96752E+03
T3=	20
T4=	80
T5=	1.4596E+02
n=	variabel
p=	5
N-table=	4

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
19L	7.2850	4.683	4		-1.37	1.50		
46L	9.5500	2.313	4		-0.13	0.74		
12L	9.8250	3.238	4		0.02	1.04		
56L	9.8333	3.249	4		0.03	1.04		
4L	12.4250	0.574	4		1.45	0.18		
Tot.gem	9.784	2.811 ng/kg		*1%-level:*	1.72	(1.73)		5
Tot.std=	1.822	1.511		*5%-level:*	1.57	(1.53)		

End Result:

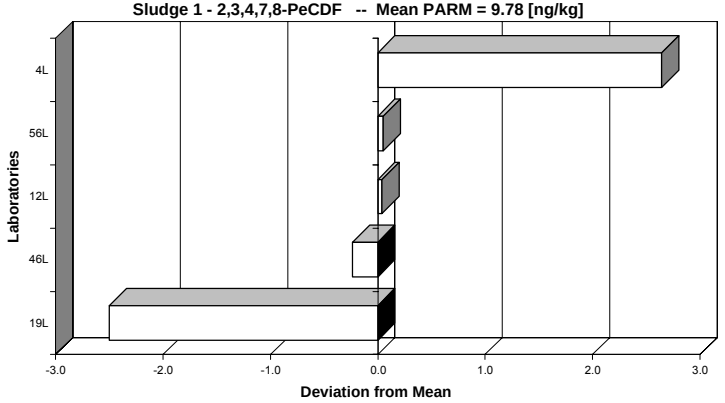
PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
7.2850	4.6834		4	3	-2.50
9.5500	2.3130		4	3	-0.23
9.8250	3.2377		4	3	0.04
9.8333	3.2494		4	3	0.05
12.4250	0.5737		4	3	2.64
9.7837		()	5	4	

RESULTS: **Mean =** **9.78367** *ng/kg*

Repeatability variance	**S2r =**	9.73094				
Repeatability std.	**Sr =**	**3.11945**	-->	31.88%	**r =**	8.7344
Between lab variance	**S2L =**	0.88695				
Reproducibility var.	**S2R =**	10.61789				
Reproducibility std.	**SR =**	**3.25851**	-->	33.31%	**R =**	9.1238

Remarks: **none**

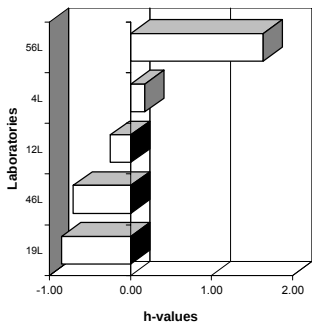
Sludge 1 - 2,3,4,7,8-PeCDF -- Mean PARM = 9.78 [ng/kg]



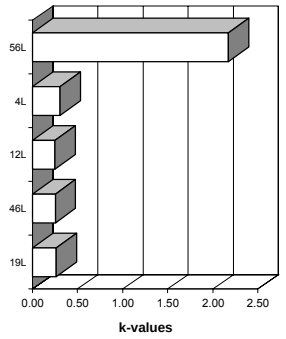
Sample: **Sludge 1**

Element: **2,3,7,8-TCDF**

Mandel's h statistics
(Sludge 1 - 2,3,7,8-TCDF)



Mandel's k statistics
(Sludge 1 - 2,3,7,8-TCDF)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - 2,3,7,8-TCDF)
Mandel's h statistics (Sludge 1 - 2,3,7,8-TCDF)
Sludge 1 - 2,3,7,8-TCDF -- Mean PARM = 10.03 [ng/kg]

	General calc.parm.
T1=	1.60521E+02
T2=	1.63166E+03
T3=	16
T4=	64
T5=	5.2140E+00
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*
19L	8.7725	0.642	4		-0.85	0.26			
46L	9.1750	0.624	4		-0.71	0.25			
12L	10.4827	0.608	4		-0.25	0.25			
4L	11.7000	0.753	4		0.17	0.31			
56L	15.8750	5.319	4	!	1.64	2.17	!!	Fail	
Tot.gem	11.201	1.589	ng/kg		*1%-level:*	1.72	(1.73)		4
Tot.std=	2.855	2.086			*5%-level:*	1.57	(1.53)		1

End Result:

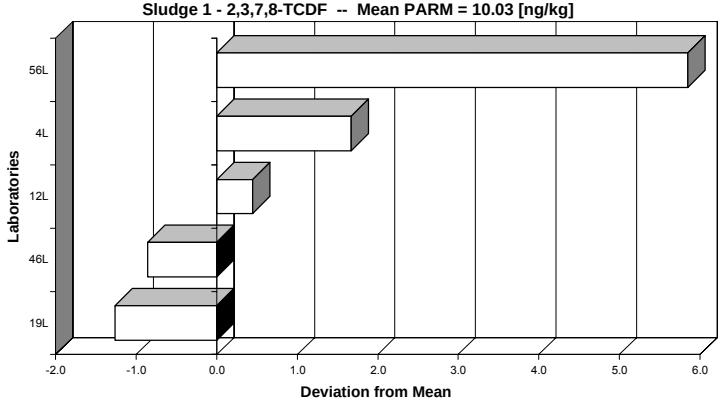
PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
8.7725	0.6422		4	3	-1.26
9.1750	0.6238		4	3	-0.86
10.4827	0.6081		4	3	0.45
11.7000	0.7528		4	3	1.67
-	-	,56L	-	-	5.84
10.0325		(56L)		4	3

RESULTS: **Mean = 10.03255** *ng/kg*

Repeatability variance	**S2r =**	0.43450				
Repeatability std.	**Sr =**	**0.65916**	-->	6.57%	**r =**	1.8457
Between lab variance	**S2L =**	1.66009				
Reproducibility var.	**S2R =**	2.09459				
Reproducibility std.	**SR =**	**1.44727**	-->	14.43%	**R =**	4.0524

Remarks: **1 Lab rejected! (56L)**

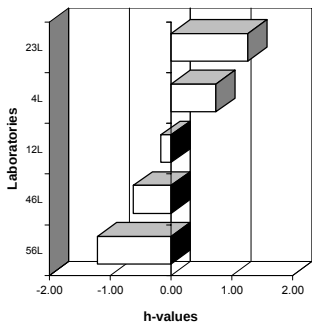
Sludge 1 - 2,3,7,8-TCDF -- Mean PARM = 10.03 [ng/kg]



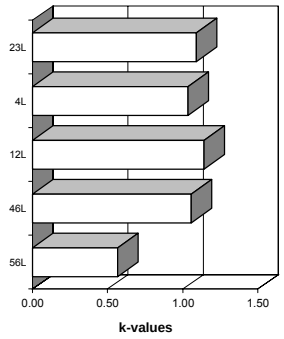
Sample: **Compost 1**

Element: **OCDD**

Mandel's h statistics (Compost 1 - OCDD)



Mandel's k statistics (Compost 1 - OCDD)



Unit: ng/kg

Mandel's k statistics (Compost 1 - OCDD)
Mandel's h statistics (Compost 1 - OCDD)
Compost 1 - OCDD -- Mean PARM = 1031 [ng/kg]

	General calc.parm.
T1=	2.06128E+04
T2=	2.42164E+07
T3=	20
T4=	80
T5=	1.9961E+05
n=	variabel
p=	5
N-table=	4

Mandel's statistics — End Result:

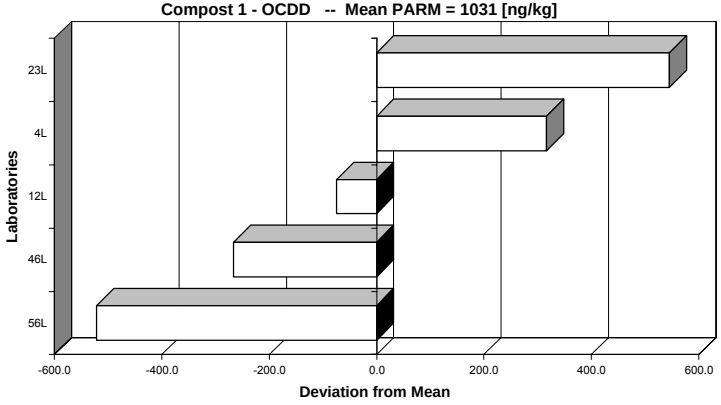
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
56L	510.2750	65.527	4		-1.21	0.57		Fail	510.2750	65.5274		4	3	-520.37
46L	765.0000	121.792	4		-0.62	1.06		Fail	765.0000	121.7922		4	3	-265.64
12L	956.3812	131.426	4		-0.17	1.14		Fail	956.3812	131.4264		4	3	-74.26
4L	1346.5500	119.590	4		0.73	1.04	Fail		1346.5500	119.5901		4	3	315.91
23L	1575.0000	125.831	4		1.26	1.09	Fail		1575.0000	125.8306		4	3	544.36
Tot.gem	1030.641	112.833 ng/kg		*1%-level:*	1.72	(1.73)		5	*1030.6412*	()		5	4	
Tot.std=	430.983	26.825		*5%-level:*	1.57	(1.53)								

RESULTS: **Mean = 1030.64124** *ng/kg*

Repeatability variance	**S2r =**	13307.04038			
Repeatability std.	**Sr =**	**115.35615**	-->	11.19%	**r =** 322.9972
Between lab variance	**S2L =**	182419.65199			
Reproducibility var.	**S2R =**	195726.69237			
Reproducibility std.	**SR =**	**442.41010**	-->	42.93%	**R =** 1238.7483

Remarks: **none**

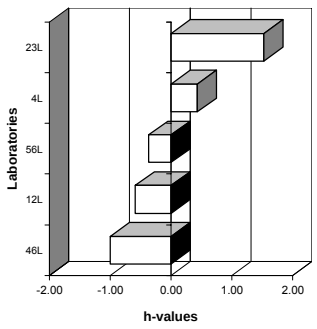
Compost 1 - OCDD -- Mean PARM = 1031 [ng/kg]



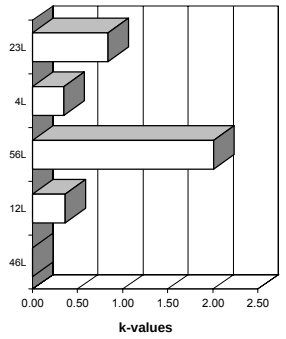
Sample: **Compost 1**

Element: **OCDF**

Mandel's h statistics (Compost 1 - OCDF)



Mandel's k statistics (Compost 1 - OCDF)



Unit: ng/kg

Mandel's k statistics (Compost 1 - OCDF)
Mandel's h statistics (Compost 1 - OCDF)
Compost 1 - OCDF -- Mean PARM = 27.54 [ng/kg]

	General calc.parm.
T1=	2.75390E+02
T2=	7.92465E+03
T3=	10
T4=	20
T5=	8.9112E+01
n=	variabel
p=	*5*
N-table=	*2*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
46L	21.0000		2		-1.00			
12L	23.7055	1.540	2		-0.59	0.36		
56L	25.1397	8.487	2		-0.37	2.01	!	
4L	30.3500	1.485	2		0.43	0.35		
23L	37.5000	3.536	2		1.53	0.84		Fail
Tot.gem	27.539	3.010 ng/kg		*1%-level:*	1.72	(2.05)		5
Tot.std=	6.526	3.310		*5%-level:*	1.57	(1.81)		

End Result:

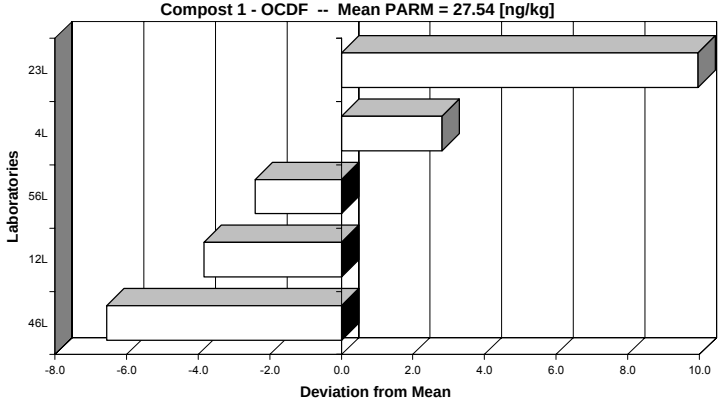
PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
21.0000			2	1	-6.54
23.7055	1.5399		2	1	-3.83
25.1397	8.4874		2	1	-2.40
30.3500	1.4849		2	1	2.81
37.5000	3.5355		2	1	9.96
27.5390	()		5	4	

RESULTS: **Mean =** **27.53904** *ng/kg*

Repeatability variance	**S2r =**	17.82245				
Repeatability std.	**Sr =**	**4.22166**	-->	15.33%	**r =**	11.8207
Between lab variance	**S2L =**	33.67234				
Reproducibility var.	**S2R =**	51.49480				
Reproducibility std.	**SR =**	**7.17599**	-->	26.06%	**R =**	20.0928

Remarks: **none**

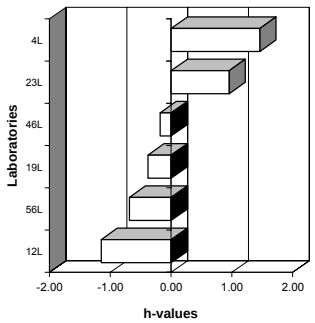
Compost 1 - OCDF -- Mean PARM = 27.54 [ng/kg]



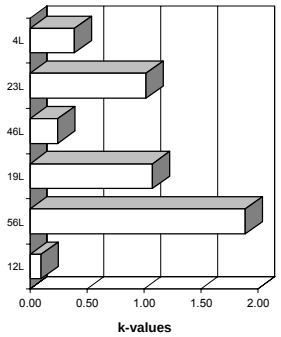
Sample: **Sludge 1**

Element: **OCDD**

Mandel's h statistics (Sludge 1 - OCDD)



Mandel's k statistics (Sludge 1 - OCDD)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - OCDD)
Mandel's h statistics (Sludge 1 - OCDD)
Sludge 1 - OCDD -- Mean PARM = 1479 [ng/kg]

	General calc.parm.
T1=	3.54869E+04
T2=	5.41109E+07
T3=	24
T4=	96
T5=	7.5285E+05
n=	variabel
p=	6
N-table=	*4*

Mandel's statistics

End Result:

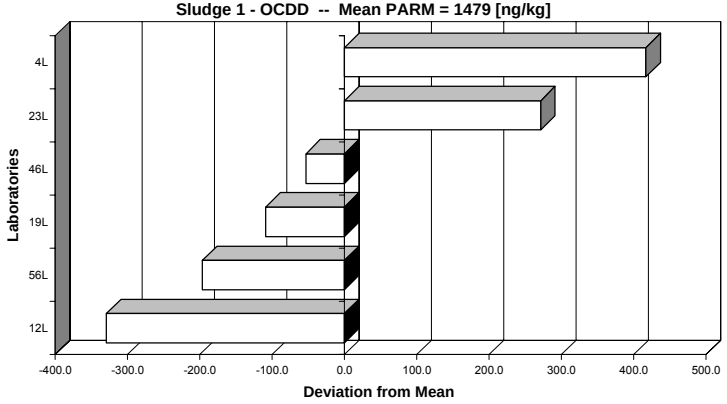
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
12L	1149.0802	20.297	4		-1.15	0.10			Fail	1149.0802	20.2969		4	3	-329.54
56L	1281.7500	387.063	4		-0.69	1.89	!!			1281.7500	387.0631		4	3	-196.87
19L	1370.0000	220.303	4		-0.38	1.08				1370.0000	220.3028		4	3	-108.62
46L	1425.0000	50.000	4		-0.19	0.24				1425.0000	50.0000		4	3	-53.62
23L	1750.0000	208.167	4		0.95	1.02				1750.0000	208.1666		4	3	271.38
4L	1895.9000	79.712	4		1.46	0.39		Fail		1895.9000	79.7116		4	3	417.28
Tot.gem	1478.622	160.924	ng/kg	*1%-level:*	1.87	(1.77)			6	*1478.6217*	()		6	5	
Tot.std=	286.286	138.255		*5%-level:*	1.66	(1.54)									

RESULTS: **Mean =** **1478.62170** *ng/kg*

Repeatability variance	**S2r =**	41825.07246				
Repeatability std.	**Sr =**	**204.51179**	-->	13.83%	**r =**	572.6330
Between lab variance	**S2L =**	71503.20658				
Reproducibility var.	**S2R =**	113328.27904				
Reproducibility std.	**SR =**	**336.64266**	-->	22.77%	**R =**	942.5994

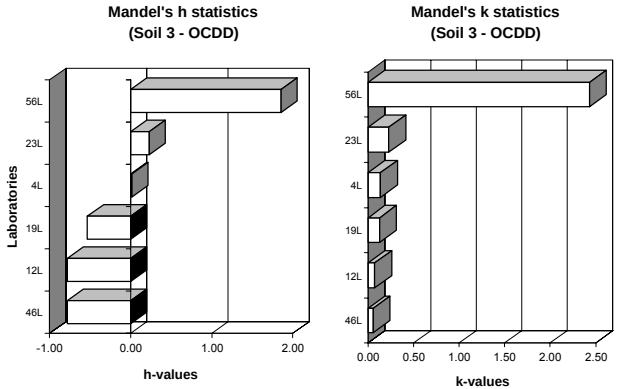
Remarks: **none**

Sludge 1 - OCDD -- Mean PARM = 1479 [ng/kg]



Sample: **Soil 3**

Element: **OCDD**



Unit: ng/kg

Mandel's k statistics (Soil 3 - OCDD)
Mandel's h statistics (Soil 3 - OCDD)
Soil 3 - OCDD -- Mean PARM = 56.89 [ng/kg]

General calc.parm.	
T1=	1.08561E+03
T2=	6.49269E+04
T3=	19
T4=	73
T5=	1.1791E+02
n=	variabel
p=	5
N-table=	4

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*
46L	45.0000	1.155	4		-0.78	0.06			
12L	45.0026	1.524	4		-0.78	0.07			
19L	52.1667	2.650	3		-0.53	0.13			
4L	68.0250	2.837	4		0.01	0.14			
23L	74.2500	4.787	4		0.23	0.23			
56L	121.1750	50.812	4	!	1.85	2.43	!!	Fail	-
Tot.gem	67.603	10.627	ng/kg		*1%-level:*	1.87	(1.77)		5
Tot.std=	28.894	19.727			*5%-level:*	1.66	(1.54)		1

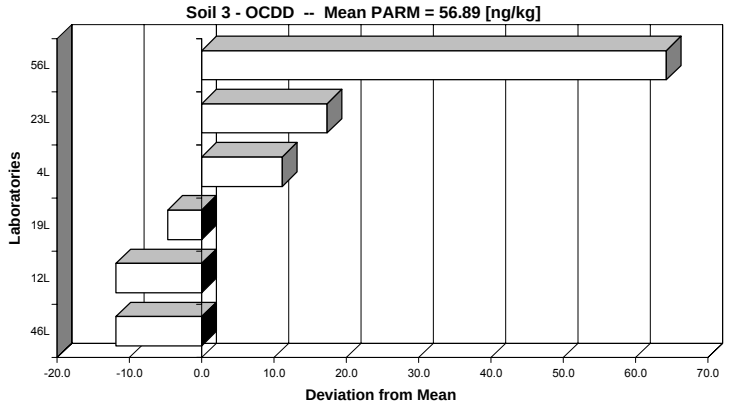
End Result:

PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
45.0000	1.1547		4	3	-11.89
45.0026	1.5236		4	3	-11.89
52.1667	2.6502		3	2	-4.72
68.0250	2.8371		4	3	11.14
74.2500	4.7871		4	3	17.36
-	-	,56L	-	-	64.29
56.8888		(56L)	5	4	

RESULTS: **Mean =** **56.88884** *ng/kg*

Repeatability variance	**S2r =**	8.42204				
Repeatability std.	**Sr =**	**2.90208**	-->	5.10%	**r =**	8.1258
Between lab variance	**S2L =**	188.96055				
Reproducibility var.	**S2R =**	197.38259				
Reproducibility std.	**SR =**	**14.04929**	-->	24.70%	**R =**	39.3380

Remarks: **1 Lab rejected! (56L)**



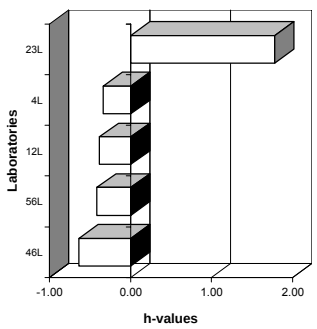
Sample: **Compost 1**

Element: **OCDF**

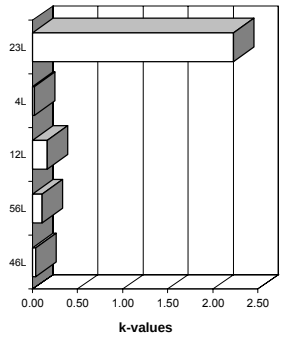
Unit: ng/kg

Mandel's k statistics (Compost 1 - OCDF)
Mandel's h statistics (Compost 1 - OCDF)
Compost 1 - OCDF -- Mean PARM = 26.4 [ng/kg]

Mandel's h statistics (Compost 1 - OCDF)



Mandel's k statistics (Compost 1 - OCDF)



General calc.parm.	
T1=	4.22325E+02
T2=	1.14161E+04
T3=	16
T4=	64
T5=	4.9164E+02
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
46L	19.5000	2.380	4		-0.64	0.04		
56L	27.3802	6.858	4		-0.42	0.11		
12L	28.5762	10.444	4		-0.38	0.16		
4L	30.1250	1.452	4		-0.34	0.02		
23L	106.0000	142.738	4	!!	1.78	2.23	!!	
Tot.gem	42.316	32.774 ng/kg		*1%-level:*	1.72	(1.73)		4
Tot.std=	35.835	61.577		*5%-level:*	1.57	(1.53)		1

End Result:

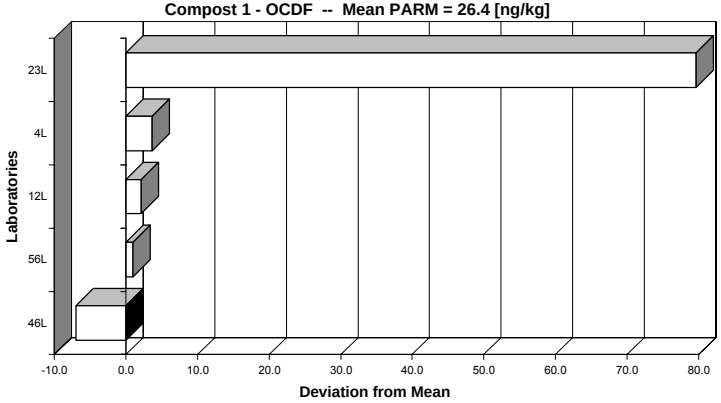
PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
19.5000	2.3805		4	3	-6.90
27.3802	6.8578		4	3	0.98
28.5762	10.4439		4	3	2.18
30.1250	1.4523		4	3	3.73
-	-	,23L	-	-	79.60
26.3953		(23L)	4	3	

RESULTS: **Mean =** **26.39533** *ng/kg*

Repeatability variance	**S2r =**	40.96990				
Repeatability std.	**Sr =**	**6.40077**	-->	24.25%	**r =**	17.9222
Between lab variance	**S2L =**	12.15150				
Reproducibility var.	**S2R =**	53.12140				
Reproducibility std.	**SR =**	**7.28844**	-->	27.61%	**R =**	20.4076

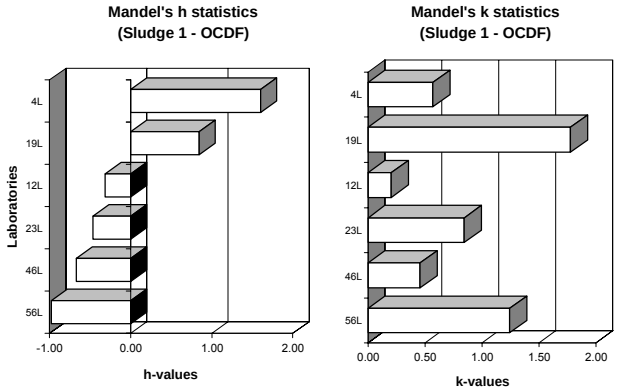
Remarks: **1 Lab rejected! (23L)**

Compost 1 - OCDF -- Mean PARM = 26.4 [ng/kg]



Sample: **Sludge 1**

Element: **OCDF**



Unit: ng/kg

Mandel's k statistics (Sludge 1 - OCDF)
Mandel's h statistics (Sludge 1 - OCDF)
Sludge 1 - OCDF -- Mean PARM = 197 [ng/kg]

	General calc.parm.
T1=	4.72719E+03
T2=	9.57403E+05
T3=	24
T4=	96
T5=	1.3498E+04
n=	variabel
p=	6
N-table=	*4*

Mandel's statistics

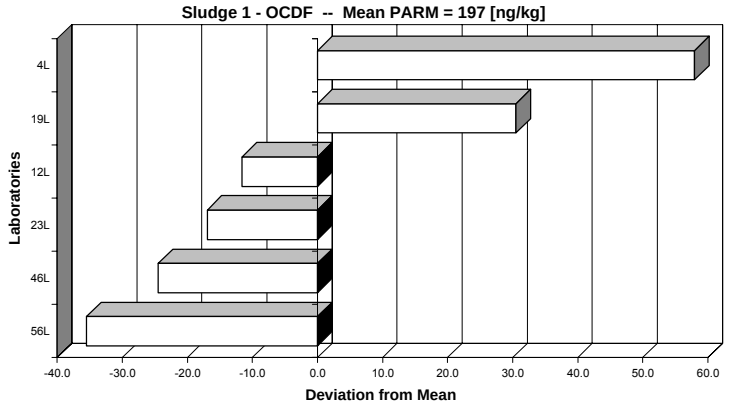
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
56L	161.4505	34.053	4		-0.98	1.24			Fail	161.4505	34.0534		4	3	-35.52
46L	172.5000	12.583	4		-0.67	0.46				172.5000	12.5831		4	3	-24.47
23L	180.0000	23.094	4		-0.47	0.84				180.0000	23.0940		4	3	-16.97
12L	185.3981	5.617	4		-0.32	0.21				185.3981	5.6169		4	3	-11.57
19L	227.5000	48.720	4		0.84	1.78	!!			227.5000	48.7203		4	3	30.53
4L	254.9500	15.587	4		1.60	0.57		Fail		254.9500	15.5875		4	3	57.98
Tot.gem	196.966	23.276	ng/kg	*1%-level:*	1.87	(1.77)			6	*196.9664*	()		6	5	
Tot.std=	36.266	15.804		*5%-level:*	1.66	(1.54)									

End Result:

RESULTS: **Mean =** **196.96644** *ng/kg*

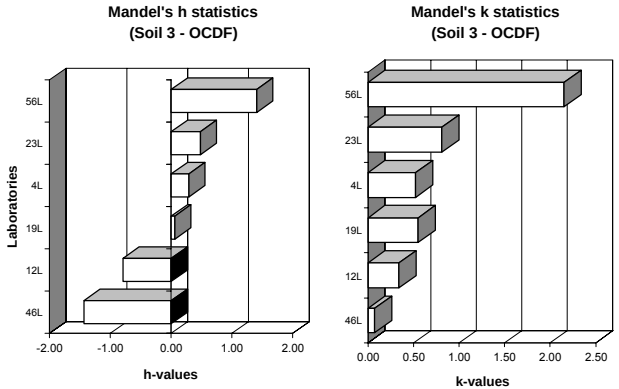
Repeatability variance	**S2r =**	749.91486				
Repeatability std.	**Sr =**	**27.38457**	-->	13.90%	**r =**	76.6768
Between lab variance	**S2L =**	1127.73420				
Reproducibility var.	**S2R =**	1877.64907				
Reproducibility std.	**SR =**	**43.33185**	-->	22.00%	**R =**	121.3292

Remarks: **none**



Sample: **Soil 3**

Element: **OCDF**



Unit: ng/kg

Mandel's k statistics (Soil 3 - OCDF)
Mandel's h statistics (Soil 3 - OCDF)

Soil 3 - OCDF -- Mean PARM = 42.27 [ng/kg]

	General calc.parm.
T1=	7.99768E+02
T2=	3.47346E+04
T3=	19
T4=	73
T5=	2.1843E+02
n=	variabel
p=	5
N-table=	4

Mandel's statistics

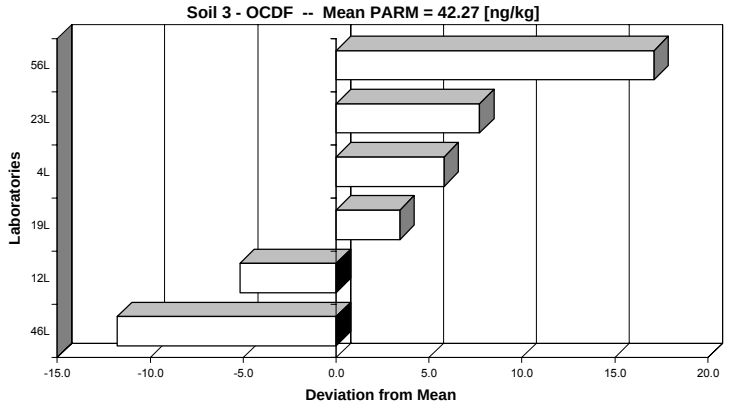
End Result:

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
46L	30.5000	0.577	4		-1.44	0.08			Fail	30.5000	0.5774		4	3	-11.77
12L	37.0921	2.586	4		-0.79	0.34				37.0921	2.5857		4	3	-5.18
19L	45.7000	4.204	3		0.06	0.55				45.7000	4.2036		3	2	3.43
4L	48.0750	4.001	4		0.29	0.53				48.0750	4.0011		4	3	5.80
23L	50.0000	6.164	4		0.48	0.81				50.0000	6.1644		4	3	7.73
56L	59.3776	16.396	4		1.41	2.15	!!	Fail		-	-	,56L	-	-	17.10
Tot.gem	45.124	5.655	ng/kg		*1%-level:*	1.87	(1.77)			5	*42.2734*	(56L)	5	4	
Tot.std=	10.143	5.580			*5%-level:*	1.66	(1.54)			1					

RESULTS: **Mean = 42.27342** *ng/kg*

Repeatability variance	**S2r =**	15.60183				
Repeatability std.	**Sr =**	**3.94992**	-->	9.34%	**r =**	11.0598
Between lab variance	**S2L =**	66.46505				
Reproducibility var.	**S2R =**	82.06688				
Reproducibility std.	**SR =**	**9.05908**	-->	21.43%	**R =**	25.3654

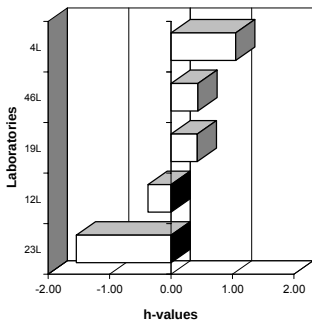
Remarks: **1 Lab rejected! (56L)**



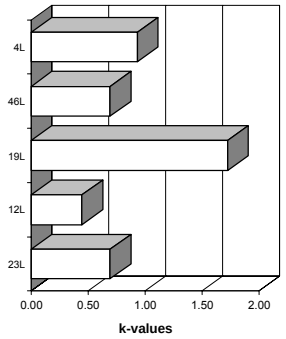
Sample: **Sludge 1**

Element: **DL PCB 77**

Mandel's h statistics
(Sludge 1 - DL PCB 77)



Mandel's k statistics
(Sludge 1 - DL PCB 77)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 77)
Mandel's h statistics (Sludge 1 - DL PCB 77)
Sludge 1 - DL PCB 77 -- Mean PARM = 6.67 [ng/kg]

	General calc.parm.
T1=	1.26359E+02
T2=	8.48262E+02
T3=	19
T4=	73
T5=	1.5623E+00
n=	variabel
p=	5
N-table=	*4*

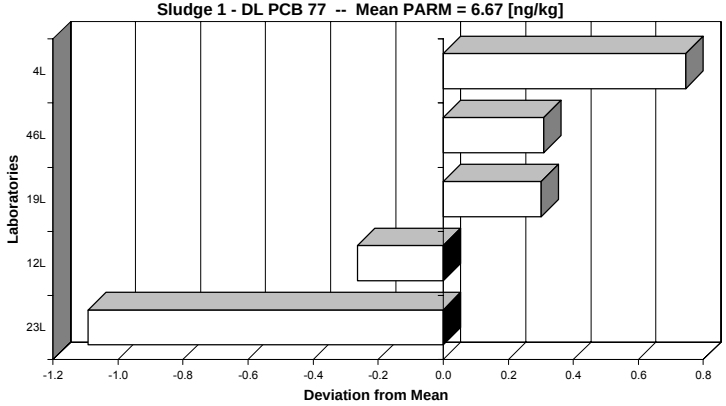
Mandel's statistics

End Result:

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
23L	5.5750	0.250	4		-1.54	0.69		Fail	5.5750	0.2500		4	3	-1.09
12L	6.4025	0.161	4		-0.37	0.45			6.4025	0.1611		4	3	-0.26
19L	6.9667	0.621	3		0.42	1.72	!		6.9667	0.6208		3	2	0.30
46L	6.9750	0.250	4		0.44	0.69			6.9750	0.2500		4	3	0.31
4L	7.4123	0.336	4		1.05	0.93		Fail	7.4123	0.3359		4	3	0.75
Tot.gem	6.666	0.324 ng/kg		*1%-level:*	1.72	(1.73)			5	*6.6663*	()	5	4	
Tot.std=	0.708	0.177		*5%-level:*	1.57	(1.53)								

RESULTS:	**Mean =**	**6.66628**	*ng/kg*			
Repeatability variance	**S2r =**	0.11159				
Repeatability std.	**Sr =**	**0.33405**	-->	5.01%	**r =**	0.9353
Between lab variance	**S2L =**	0.49272				
Reproducibility var.	**S2R =**	0.60431				
Reproducibility std.	**SR =**	**0.77737**	-->	11.66%	**R =**	2.1766
Remarks:	**none**					

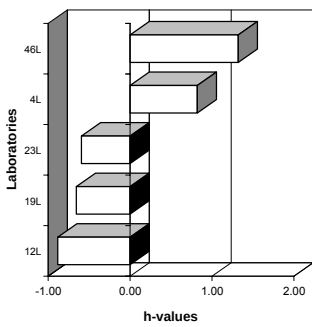
Sludge 1 - DL PCB 77 -- Mean PARM = 6.67 [ng/kg]



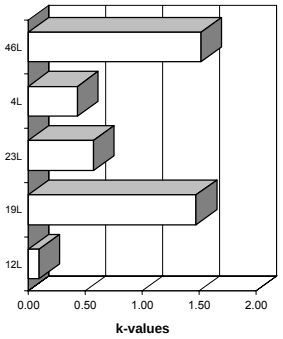
Sample: **Sludge 1**

Element: **DL PCB 81**

Mandel's h statistics
(Sludge 1 - DL PCB 81)



Mandel's k statistics
(Sludge 1 - DL PCB 81)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 81)
Mandel's h statistics (Sludge 1 - DL PCB 81)
Sludge 1 - DL PCB 81 -- Mean PARM = 0.524 [ng/kg]

	General calc.parm.
T1=	1.01193E+01
T2=	6.31774E+00
T3=	19
T4=	73
T5=	4.2526E-02
n=	variabel
p=	5
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
12L	0.3083	0.006	4		-0.88	0.10		Fail
19L	0.3627	0.085	3		-0.66	1.47		Fail
23L	0.3775	0.033	4		-0.60	0.57		Fail
4L	0.7246	0.025	4		0.82	0.43	Fail	
46L	0.8475	0.087	4		1.32	1.52	Fail	
Tot.gem	0.524	0.047 ng/kg		*1%-level:*	1.72	(1.73)		
Tot.std=	0.244	0.037		*5%-level:*	1.57	(1.53)		

End Result:

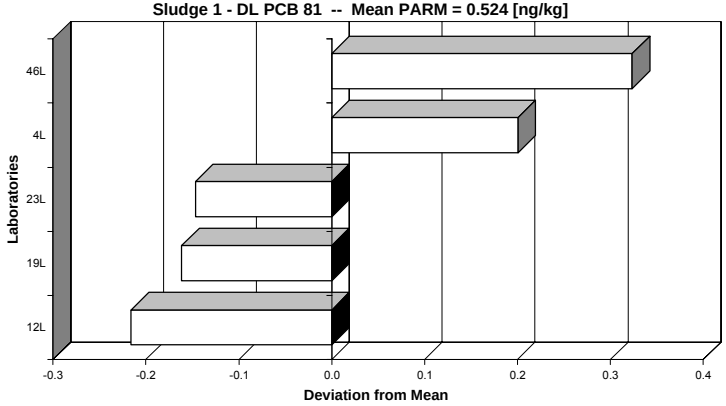
	PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
	0.3083	0.0055		4	3	-0.22
	0.3627	0.0849		3	2	-0.16
	0.3775	0.0330		4	3	-0.15
	0.7246	0.0249		4	3	0.20
	0.8475	0.0873		4	3	0.32
5	*0.5241*	()		5	4	

RESULTS: **Mean =** **0.52410** *ng/kg*

Repeatability variance	**S2r =**	0.00304				
Repeatability std.	**Sr =**	**0.05511**	-->	10.52%	**r =**	0.1543
Between lab variance	**S2L =**	0.06044				
Reproducibility var.	**S2R =**	0.06347				
Reproducibility std.	**SR =**	**0.25194**	-->	48.07%	**R =**	0.7054

Remarks: **none**

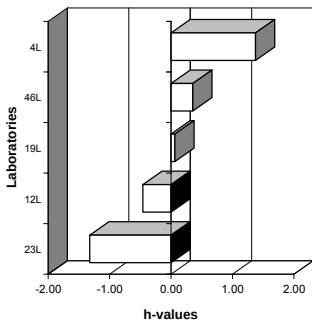
Sludge 1 - DL PCB 81 -- Mean PARM = 0.524 [ng/kg]



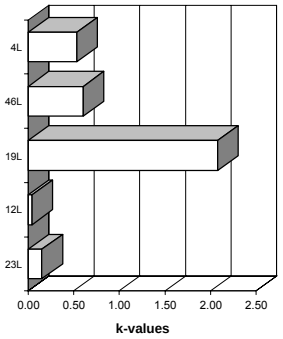
Sample: **Sludge 1**

Element: **DL PCB 105**

Mandel's h statistics
(Sludge 1 - DL PCB 105)



Mandel's k statistics
(Sludge 1 - DL PCB 105)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 105)
Mandel's h statistics (Sludge 1 - DL PCB 105)
Sludge 1 - DL PCB 105 -- Mean PARM = 19.18 [ng/kg]

	General calc.parm.
T1=	3.06948E+02
T2=	5.96805E+03
T3=	16
T4=	64
T5=	2.2105E+01
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
23L	16.2500	0.500	4		-1.33	0.15		
12L	18.2000	0.141	4		-0.46	0.04		
19L	19.3500	6.859	2		0.06	2.08	!!	
46L	20.0000	2.000	4		0.35	0.61		
4L	22.2870	1.760	4		1.38	0.53		
Tot.gem	19.217	2.252 ng/kg		*1%-level:*	1.72	(1.73)		4
Tot.std=	2.230	2.695		*5%-level:*	1.57	(1.53)		1

End Result:

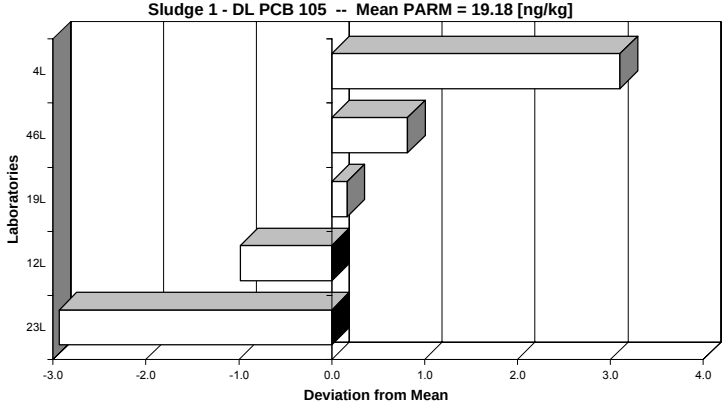
PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
16.2500	0.5000		4	3	-2.93
18.2000	0.1414		4	3	-0.98
-	-	,19L	-	-	0.17
20.0000	2.0000		4	3	0.82
22.2870	1.7602		4	3	3.10
19.1843		(19L)	4	3	

RESULTS: **Mean =** **19.18425** *ng/kg*

Repeatability variance	**S2r =**	1.84209				
Repeatability std.	**Sr =**	**1.35724**	-->	7.07%	**r =**	3.8003
Between lab variance	**S2L =**	6.16317				
Reproducibility var.	**S2R =**	8.00526				
Reproducibility std.	**SR =**	**2.82936**	-->	14.75%	**R =**	7.9222

Remarks: **1 Lab rejected! (19L)**

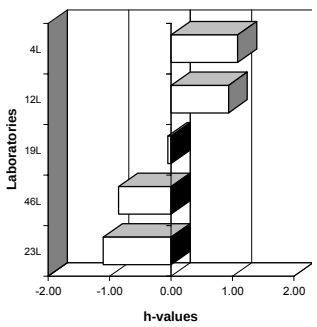
Sludge 1 - DL PCB 105 -- Mean PARM = 19.18 [ng/kg]



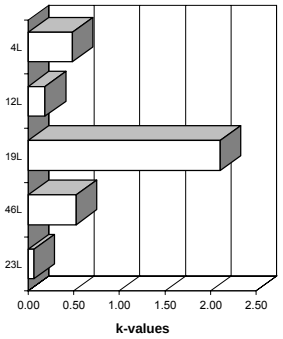
Sample: **Sludge 1**

Element: **DL PCB 114**

Mandel's h statistics
(Sludge 1 - DL PCB 114)



Mandel's k statistics
(Sludge 1 - DL PCB 114)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 114)
Mandel's h statistics (Sludge 1 - DL PCB 114)
Sludge 1 - DL PCB 114 -- Mean PARM = 1.37 [ng/kg]

General calc.parm.	
T1=	2.18990E+01
T2=	3.18006E+01
T3=	16
T4=	64
T5=	5.4269E-02
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*
23L	0.9900	0.012	4		-1.11	0.06	
46L	1.0750	0.096	4		-0.85	0.53	
19L	1.3450	0.382	4		-0.06	2.11	!!
12L	1.6800	0.034	4		0.93	0.19	
4L	1.7298	0.087	4		1.08	0.48	
Tot.gem	1.364	0.122	ng/kg		*1%-level:*	1.72	(1.73)
Tot.std=	0.338	0.149			*5%-level:*	1.57	(1.53)

End Result:

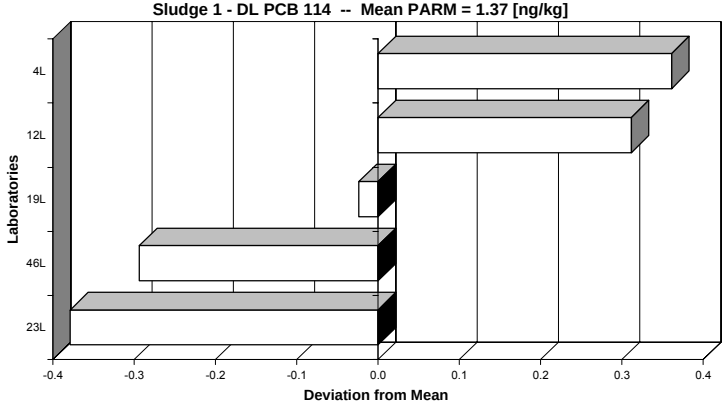
\vX > AvST+2std	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
	Fail	0.9900	0.0115		4	3	-0.38
		1.0750	0.0957		4	3	-0.29
-	-	-	-	,19L	-	-	-0.02
	Fail	1.6800	0.0337		4	3	0.31
	Fail	1.7298	0.0875		4	3	0.36
	4	*1.3687*		(19L)	4	3	
	1						

RESULTS: **Mean =** **1.36869** *ng/kg*

Repeatability variance	**S2r =**	0.00452				
Repeatability std.	**Sr =**	**0.06725**	-->	4.91%	**r =**	0.1883
Between lab variance	**S2L =**	0.15118				
Reproducibility var.	**S2R =**	0.15570				
Reproducibility std.	**SR =**	**0.39459**	-->	28.83%	**R =**	1.1049

Remarks: **1 Lab rejected! (19L)**

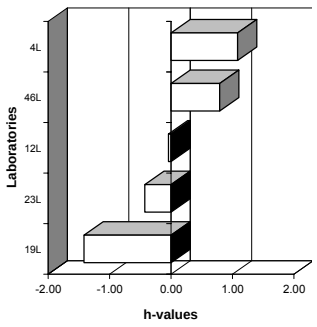
Sludge 1 - DL PCB 114 -- Mean PARM = 1.37 [ng/kg]



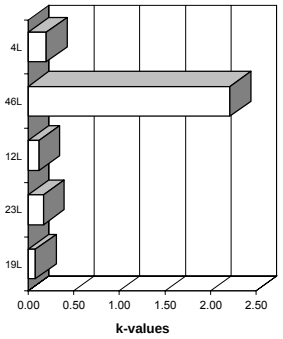
Sample: **Sludge 1**

Element: **DL PCB 118**

Mandel's h statistics
(Sludge 1 - DL PCB 118)



Mandel's k statistics
(Sludge 1 - DL PCB 118)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 118)
Mandel's h statistics (Sludge 1 - DL PCB 118)
Sludge 1 - DL PCB 118 -- Mean PARM = 32.59 [ng/kg]

	General calc.parm.
T1=	5.21365E+02
T2=	1.71649E+04
T3=	16
T4=	64
T5=	8.5628E+00
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
19L	28.0750	0.457	4		-1.42	0.08		
23L	31.7500	0.957	4		-0.42	0.17		
12L	33.1750	0.670	4		-0.04	0.12		
46L	36.2500	12.527	4		0.79	2.22	!!	
4L	37.3413	1.131	4		1.09	0.20		
Tot.gem	33.318	3.149 ng/kg		*1%-level:*	1.72	(1.73)		4
Tot.std=	3.700	5.249		*5%-level:*	1.57	(1.53)		1

End Result:

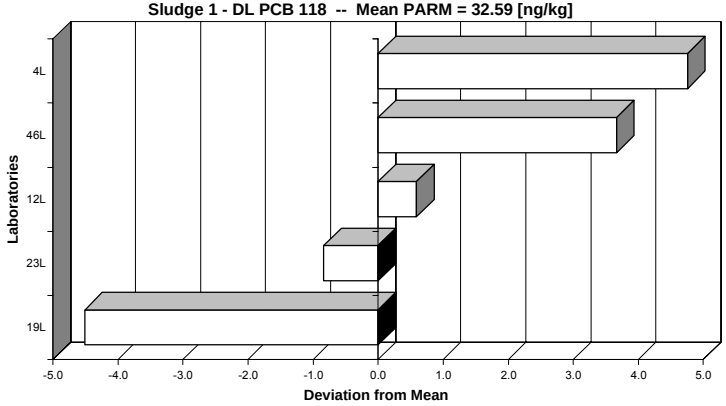
PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
28.0750	0.4573		4	3	-4.51
31.7500	0.9574		4	3	-0.84
33.1750	0.6702		4	3	0.59
-	-	,46L	-	-	3.66
37.3413	1.1310		4	3	4.76
32.5853		(46L)	4	3	

RESULTS: **Mean =** **32.58531** *ng/kg*

Repeatability variance	**S2r =**	0.71356				
Repeatability std.	**Sr =**	**0.84473**	-->	2.59%	**r =**	2.3652
Between lab variance	**S2L =**	14.49072				
Reproducibility var.	**S2R =**	15.20429				
Reproducibility std.	**SR =**	**3.89927**	-->	11.97%	**R =**	10.9179

Remarks: **1 Lab rejected! (46L)**

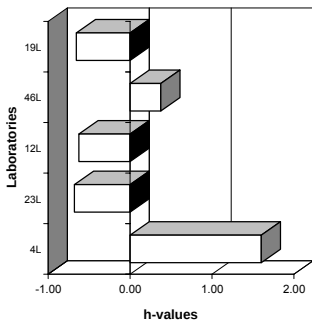
Sludge 1 - DL PCB 118 -- Mean PARM = 32.59 [ng/kg]



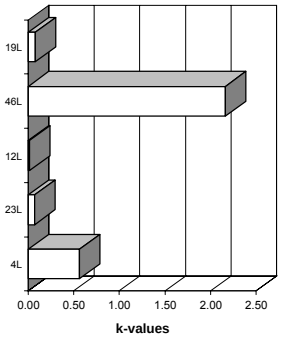
Sample: **Sludge 1**

Element: **DL PCB 123**

Mandel's h statistics
(Sludge 1 - DL PCB 123)



Mandel's k statistics
(Sludge 1 - DL PCB 123)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 123)
Mandel's h statistics (Sludge 1 - DL PCB 123)

#NUM!

General calc.parm.

T1=	#NUM!
T2=	#NUM!
T3=	
T4=	
T5=	
n=	variabel
p=	
N-table=	*4*

Mandel's statistics

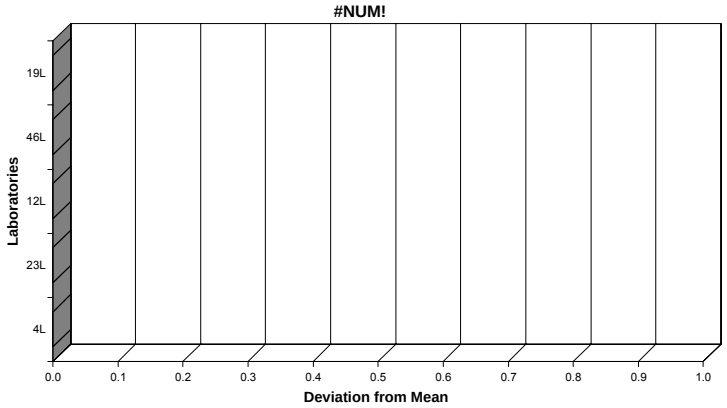
End Result:

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
4L	4.6928	0.458	4	!	1.60	0.56	Fail	-	-	,4L	-	-		#NUM!
23L	0.7575	0.058	4		-0.68	0.07		#NUM!	-	,23L	-	-		#NUM!
12L	0.8498	0.013	4		-0.63	0.02		-	-	,12L	-	-		#NUM!
46L	2.5750	1.752	4		0.37	2.16	!!	-	-	,46L	-	-		#NUM!
19L	0.8003	0.065	4		-0.66	0.08		-	-	,19L	-	-		#NUM!
Tot.gem	1.935	0.469 ng/kg		*1%-level:*	1.72	(1.73)			*#NUM!*		(4L,23L,12L,46L,19L)		-1	
Tot.std=	1.722	0.739		*5%-level:*	1.57	(1.53)		5						

RESULTS: **Mean =** **#NUM!** *ng/kg*

Repeatability variance	**S2r =**	#DIV/0!				
Repeatability std.	**Sr =**	**#DIV/0!**	-->	#DIV/0!	**r =**	#DIV/0!
Between lab variance	**S2L =**	#NUM!				
Reproducibility var.	**S2R =**	#NUM!				
Reproducibility std.	**SR =**	**#NUM!**	-->	#NUM!	**R =**	#NUM!

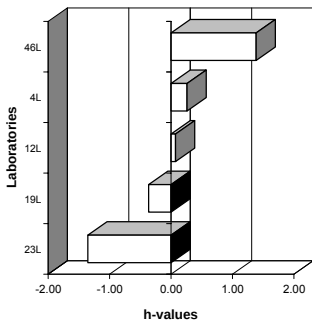
Remarks: **5 Labs rejected! (4L,23L,12L,46L,19L)**



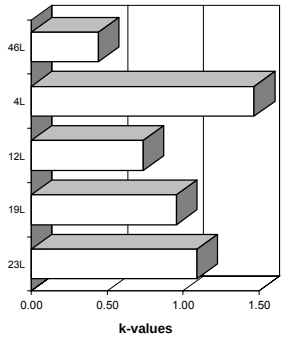
Sample: **Sludge 1**

Element: **DL PCB 126**

Mandel's h statistics
(Sludge 1 - DL PCB 126)



Mandel's k statistics
(Sludge 1 - DL PCB 126)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 126)
Mandel's h statistics (Sludge 1 - DL PCB 126)
Sludge 1 - DL PCB 126 -- Mean PARM = 0.25 [ng/kg]

	General calc.parm.
T1=	4.77910E+00
T2=	1.29646E+00
T3=	19
T4=	73
T5=	1.1401E-02
n=	variabel
p=	5
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*
23L	0.1450	0.031	4		-1.36	1.09	
19L	0.2220	0.027	3		-0.36	0.96	
12L	0.2558	0.021	4		0.07	0.74	
4L	0.2700	0.042	4		0.26	1.47	
46L	0.3575	0.013	4		1.39	0.44	
Tot.gem	0.250	0.027 ng/kg		*1%-level:*	1.72	(1.73)	
Tot.std=	0.077	0.011		*5%-level:*	1.57	(1.53)	

End Result:

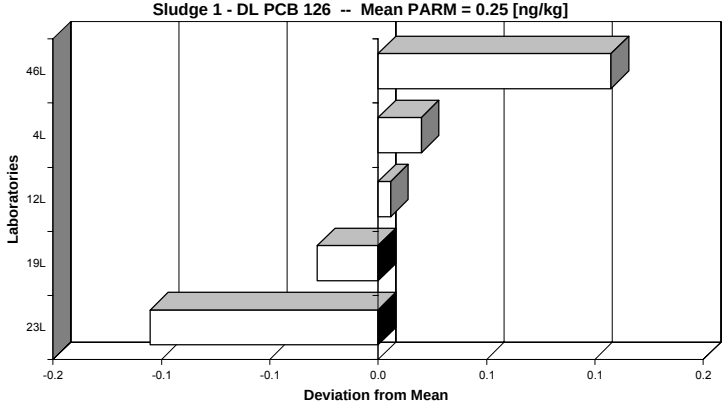
\vX > AvST+2std	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
	Fail	0.1450	0.0311		4	3	-0.11
	Fail	0.2220	0.0272		3	2	-0.03
		0.2558	0.0209		4	3	0.01
		0.2700	0.0418		4	3	0.02
Fail		0.3575	0.0126		4	3	0.11
	5	*0.2501*	()		5	4	

RESULTS: **Mean =** **0.25006** *ng/kg*

Repeatability variance	**S2r =**	0.00081				
Repeatability std.	**Sr =**	**0.02854**	-->	11.41%	**r =**	0.0799
Between lab variance	**S2L =**	0.00601				
Reproducibility var.	**S2R =**	0.00683				
Reproducibility std.	**SR =**	**0.08261**	-->	33.04%	**R =**	0.2313

Remarks: **none**

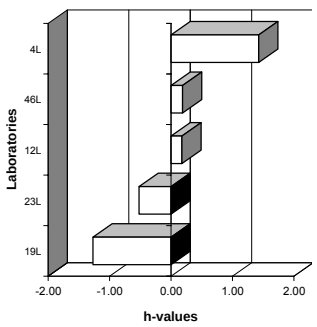
Sludge 1 - DL PCB 126 -- Mean PARM = 0.25 [ng/kg]



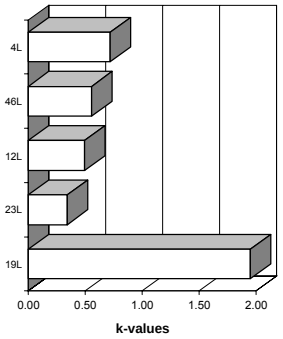
Sample: **Sludge1**

Element: **DL PCB 156**

Mandel's h statistics (Sludge1 - DL PCB 156)



Mandel's k statistics (Sludge1 - DL PCB 156)



Unit: ng/kg

Mandel's k statistics (Sludge1 - DL PCB 156)
Mandel's h statistics (Sludge1 - DL PCB 156)

Sludge1 - DL PCB 156 -- Mean PARM = 7.31 [ng/kg]

	General calc.parm.
T1=	1.09765E+02
T2=	8.08898E+02
T3=	15
T4=	57
T5=	7.3657E-01
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*
19L	5.9575	0.927	4		-1.27	1.95	!!		Fail
23L	6.6000	0.163	4		-0.52	0.34			
12L	7.1875	0.234	4		0.17	0.49			
46L	7.2000	0.265	3		0.19	0.56			
4L	8.2538	0.343	4		1.43	0.72		Fail	
Tot.gem	7.040	0.386	ng/kg	*1%-level:*	1.72	(1.73)			4
Tot.std=	0.849	0.309		*5%-level:*	1.57	(1.53)			1

End Result:

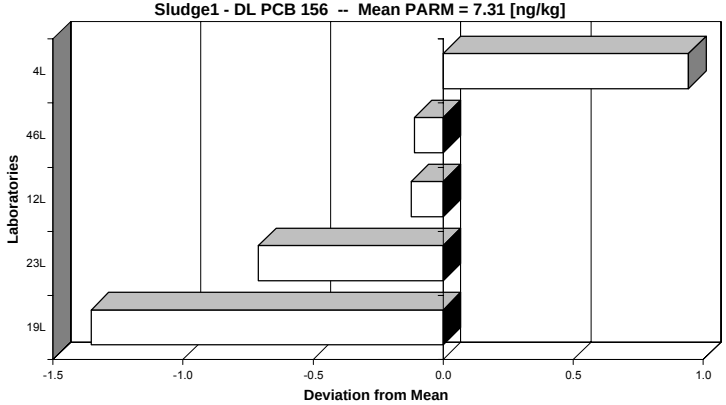
	PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
	-	-	,19L	-	-	-1.35
	6.6000	0.1633		4	3	-0.71
	7.1875	0.2341		4	3	-0.12
	7.2000	0.2646		3	2	-0.11
	8.2538	0.3426		4	3	0.94
	7.3103		(19L)	4	3	

RESULTS: **Mean =** **7.31031** *ng/kg*

Repeatability variance	**S2r =**	0.06696				
Repeatability std.	**Sr =**	**0.25877**	-->	3.54%	**r =**	0.7246
Between lab variance	**S2L =**	0.48872				
Reproducibility var.	**S2R =**	0.55568				
Reproducibility std.	**SR =**	**0.74544**	-->	10.20%	**R =**	2.0872

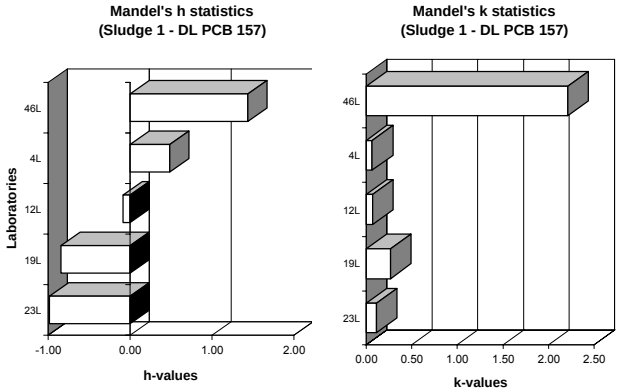
Remarks: **1 Lab rejected! (19L)**

Sludge1 - DL PCB 156 -- Mean PARM = 7.31 [ng/kg]



Sample: **Sludge 1**

Element: **DL PCB 157**



Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 157)
Mandel's h statistics (Sludge 1 - DL PCB 157)
Sludge 1 - DL PCB 157 -- Mean PARM = 0.864 [ng/kg]

	General calc.parm.
T1=	1.38191E+01
T2=	1.22413E+01
T3=	16
T4=	64
T5=	3.8332E-02
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
23L	0.7175	0.042	4		-0.99	0.11		
19L	0.7510	0.099	4		-0.84	0.27		
12L	0.9265	0.026	4		-0.09	0.07		
4L	1.0598	0.024	4		0.49	0.07		
46L	1.2800	0.814	4		1.44	2.21	!!	
Tot.gem	0.947	0.201 ng/kg		*1%-level:*	1.72	(1.73)		4
Tot.std=	0.232	0.344		*5%-level:*	1.57	(1.53)		1

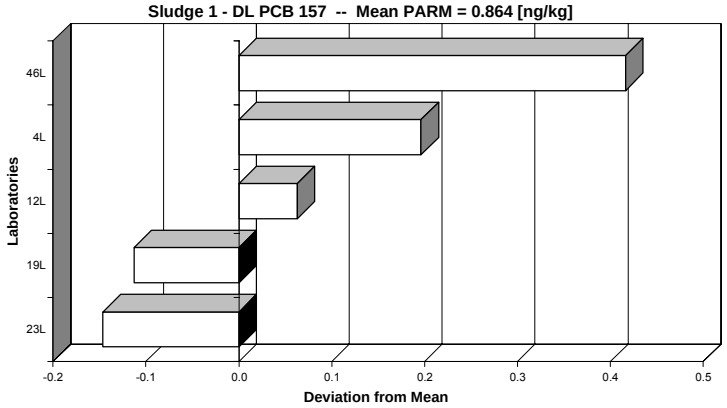
End Result:

	PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
	0.7175	0.0419		4	3	-0.15
	0.7510	0.0988		4	3	-0.11
	0.9265	0.0258		4	3	0.06
	1.0598	0.0244		4	3	0.20
-	-	,46L	-	-		0.42
	0.8637	(46L)		4	3	

RESULTS: **Mean =** **0.86369** *ng/kg*

Repeatability variance	**S2r =**	0.00319				
Repeatability std.	**Sr =**	**0.05652**	-->	6.54%	**r =**	0.1583
Between lab variance	**S2L =**	0.02469				
Reproducibility var.	**S2R =**	0.02788				
Reproducibility std.	**SR =**	**0.16699**	-->	19.33%	**R =**	0.4676

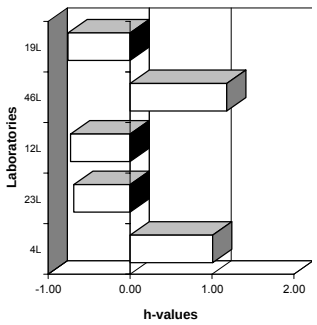
Remarks: **1 Lab rejected! (46L)**



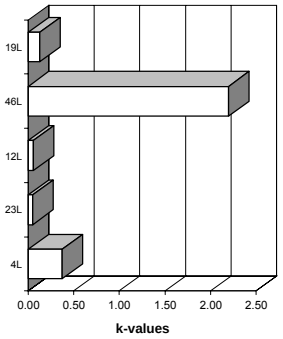
Sample: **Sludge 1**

Element: **DL PCB 167**

Mandel's h statistics
(Sludge 1 - DL PCB 167)



Mandel's k statistics
(Sludge 1 - DL PCB 167)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 167)
Mandel's h statistics (Sludge 1 - DL PCB 167)

#NUM!

General calc.parm.

T1=	#NUM!
T2=	#NUM!
T3=	
T4=	
T5=	
n=	variabel
p=	
N-table=	*4*

Mandel's statistics

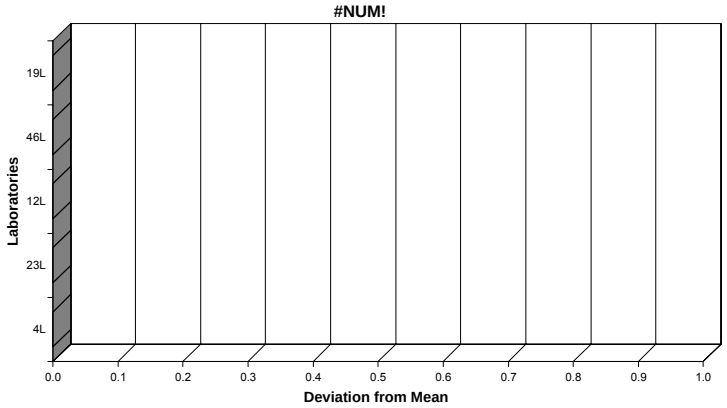
End Result:

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
4L	5.1284	0.758	4		1.01	0.37		-	-	,4L	-	-		#NUM!
23L	3.2250	0.096	4		-0.69	0.05		-	-	,23L	-	-		#NUM!
12L	3.1775	0.108	4		-0.73	0.05		-	-	,12L	-	-		#NUM!
46L	5.3250	4.451	4		1.18	2.20	!!	-	-	,46L	-	-		#NUM!
19L	3.1450	0.254	4		-0.76	0.13			#NUM!	- ,19L	-	-		#NUM!
Tot.gem	4.000	1.133 ng/kg		*1%-level:*	1.72	(1.73)			*#NUM!*		(4L,23L,12L,46L,19L)		-1	
Tot.std=	1.122	1.874		*5%-level:*	1.57	(1.53)		5						

RESULTS: **Mean =** **#NUM!** *ng/kg*

Repeatability variance	**S2r =**	#DIV/0!				
Repeatability std.	**Sr =**	**#DIV/0!**	-->	#DIV/0!	**r =**	#DIV/0!
Between lab variance	**S2L =**	#NUM!				
Reproducibility var.	**S2R =**	#NUM!				
Reproducibility std.	**SR =**	**#NUM!**	-->	#NUM!	**R =**	#NUM!

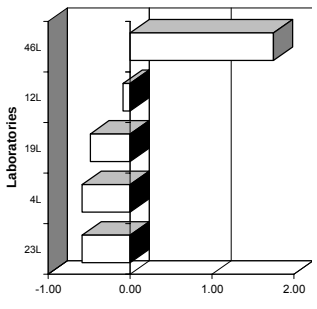
Remarks: **5 Labs rejected! (4L,23L,12L,46L,19L)**



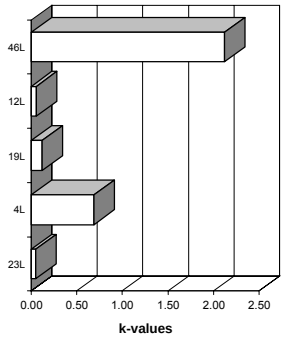
Sample: **Sludge 1**

Element: **DL PCB 189**

Mandel's h statistics
(Sludge 1 - DL PCB 189)



Mandel's k statistics
(Sludge 1 - DL PCB 189)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - DL PCB 189)
Mandel's h statistics (Sludge 1 - DL PCB 189)

#NUM!

General calc.parm.

T1=	#NUM!
T2=	#NUM!
T3=	
T4=	
T5=	
n=	variabel
p=	
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
23L	0.8900	0.038	4		-0.59	0.05		
4L	0.8909	0.506	4		-0.59	0.69		-
19L	0.9340	0.089	4		-0.49	0.12		-
12L	1.1175	0.043	4		-0.09	0.06		-
46L	1.9500	1.567	4	!!	1.75	2.12	!!	-
Tot.gem	1.156	0.449	ng/kg		*1%-level:*	1.72	(1.73)	
Tot.std=	0.453	0.655			*5%-level:*	1.57	(1.53)	

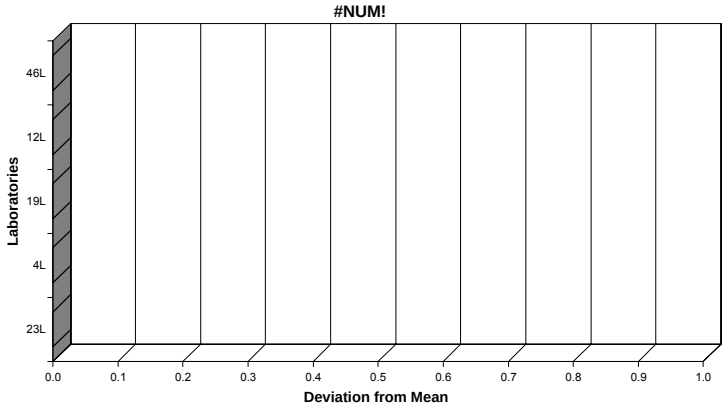
End Result:

PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
#NUM!	-	,23L	-	-	#NUM!
-	-	,4L	-	-	#NUM!
-	-	,19L	-	-	#NUM!
-	-	,12L	-	-	#NUM!
-	-	,46L	-	-	#NUM!
#NUM!		(4L,23L,12L,46L,19L)		-1	
5					

RESULTS: **Mean =** **#NUM!** *ng/kg*

Repeatability variance	**S2r =**	#DIV/0!				
Repeatability std.	**Sr =**	**#DIV/0!**	-->	#DIV/0!	**r =**	#DIV/0!
Between lab variance	**S2L =**	#NUM!				
Reproducibility var.	**S2R =**	#NUM!				
Reproducibility std.	**SR =**	**#NUM!**	-->	#NUM!	**R =**	#NUM!

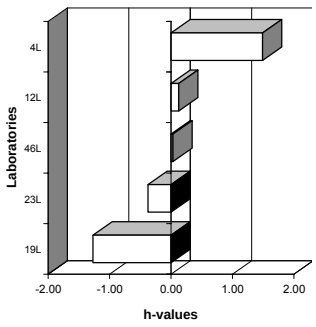
Remarks: **5 Labs rejected! (4L,23L,12L,46L,19L)**



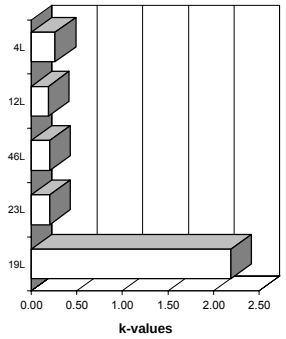
Sample: **Sludge 1**

Element: **Total DL PCB**

Mandel's h statistics
(Sludge 1 - Total DL PCB)



Mandel's k statistics
(Sludge 1 - Total DL PCB)



Unit: ng/kg

Mandel's k statistics (Sludge 1 - Total DL PCB)
Mandel's h statistics (Sludge 1 - Total DL PCB)

Sludge 1 - Total DL PCB -- Mean PARM = 75.64 [ng/kg]

General calc.parm.	
T1=	1.13809E+03
T2=	8.74948E+04
T3=	15
T4=	57
T5=	2.3213E+01
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
19L	56.3732	14.578	4		-1.27	2.19	!!	Fail
23L	67.2775	1.357	4		-0.37	0.20		
46L	72.1303	1.363	3		0.03	0.21		
12L	73.3050	1.258	4		0.13	0.19		
4L	89.8429	1.754	4		1.49	0.26		Fail
Tot.gem	71.786	4.062	ng/kg	*1%-level:*	1.72	(1.73)		4
Tot.std=	12.107	5.882		*5%-level:*	1.57	(1.53)		1

End Result:

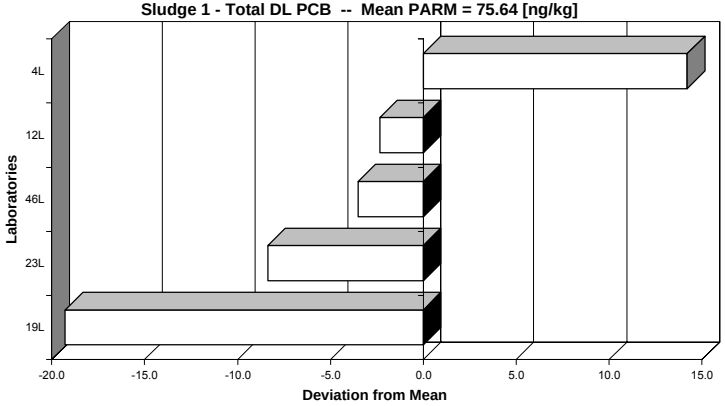
-	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
-	-	,19L	-	-		-19.27
	67.2775	1.3569		4	3	-8.36
	72.1303	1.3629		3	2	-3.51
	73.3050	1.2580		4	3	-2.33
	89.8429	1.7537		4	3	14.20
	75.6389	(19L)		4	3	

RESULTS: **Mean =** **75.63891** *ng/kg*

Repeatability variance	**S2r =**	2.11024				
Repeatability std.	**Sr =**	**1.45267**	-->	1.92%	**r =**	4.0675
Between lab variance	**S2L =**	101.62752				
Reproducibility var.	**S2R =**	103.73776				
Reproducibility std.	**SR =**	**10.18517**	-->	13.47%	**R =**	28.5185

Remarks: **1 Lab rejected! (19L)**

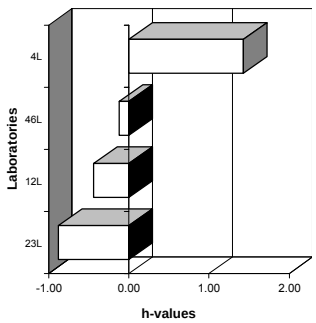
Sludge 1 - Total DL PCB -- Mean PARM = 75.64 [ng/kg]



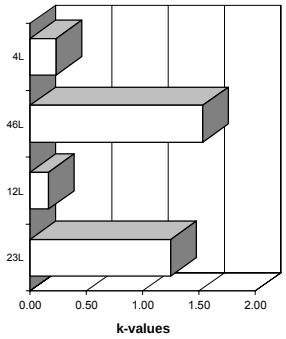
Sample: **Compost 1**

Element: **PCB 77**

Mandel's h statistics
(Compost 1 - PCB 77)



Mandel's k statistics
(Compost 1 - PCB 77)



Unit: ng/kg

Mandel's k statistics (Compost 1 - PCB 77)
Mandel's h statistics (Compost 1 - PCB 77)
Compost 1 - PCB 77 -- Mean PARM = 0.132 [ng/kg]

	General calc.parm.
T1=	2.10450E+00
T2=	2.78903E-01
T3=	16
T4=	64
T5=	1.0204E-03
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

End Result:

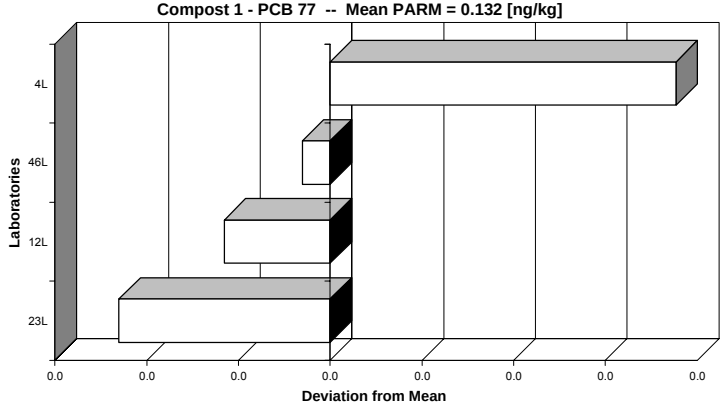
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
23L	0.1200	0.012	4		-0.87	1.25			0.1200	0.0115		4	3	-0.01
12L	0.1258	0.002	4		-0.44	0.16			0.1258	0.0015		4	3	-0.01
46L	0.1300	0.014	4		-0.12	1.53	!		0.1300	0.0141		4	3	0.00
4L	0.1504	0.002	4	!	1.43	0.23	Fail		0.1504	0.0021		4	3	0.02
Tot.gem	0.132	0.007	ng/kg		*1%-level:*	1.49	(1.67)		4	*0.1315*	()	4	3	
Tot.std=	0.013	0.006			*5%-level:*	1.42	(1.5)							

RESULTS: **Mean =** **0.13153** *ng/kg*

Repeatability variance	**S2r =**	0.00009				
Repeatability std.	**Sr =**	**0.00922**	-->	7.01%	**r =**	0.0258
Between lab variance	**S2L =**	0.00015				
Reproducibility var.	**S2R =**	0.00024				
Reproducibility std.	**SR =**	**0.01544**	-->	11.74%	**R =**	0.0432

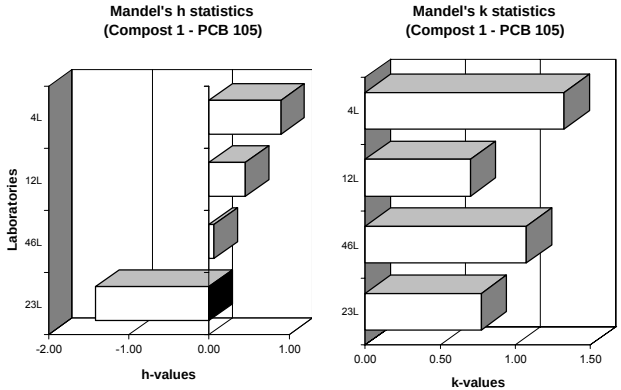
Remarks: **none**

Compost 1 - PCB 77 -- Mean PARM = 0.132 [ng/kg]



Sample: **Compost 1**

Element: **PCB 105**



Unit: ng/kg

Mandel's k statistics (Compost 1 - PCB 105)
Mandel's h statistics (Compost 1 - PCB 105)
Compost 1 - PCB 105 -- Mean PARM = 0.683 [ng/kg]

	General calc.parm.
T1=	1.09233E+01
T2=	7.63227E+00
T3=	16
T4=	64
T5=	4.7234E-02
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
23L	0.5125	0.049	4		-1.41	0.77		Fail
46L	0.6900	0.067	4		0.06	1.07		
12L	0.7375	0.044	4		0.45	0.70	Fail	
4L	0.7908	0.083	4		0.90	1.33	Fail	
Tot.gem	0.683	0.061 ng/kg		*1%-level:*	1.49	(1.67)		
Tot.std=	0.121	0.018		*5%-level:*	1.42	(1.5)		

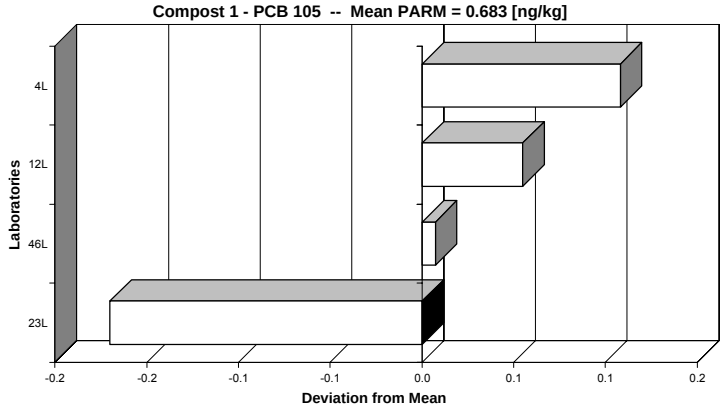
End Result:

	PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
	0.5125	0.0486		4	3	-0.17
	0.6900	0.0673		4	3	0.01
	0.7375	0.0439		4	3	0.05
	0.7908	0.0832		4	3	0.11
4	*0.6827*	()		4	3	

RESULTS: **Mean =** **0.68271** *ng/kg*

Repeatability variance	**S2r =**	0.00394				
Repeatability std.	**Sr =**	**0.06274**	-->	9.19%	**r =**	0.1757
Between lab variance	**S2L =**	0.01359				
Reproducibility var.	**S2R =**	0.01752				
Reproducibility std.	**SR =**	**0.13238**	-->	19.39%	**R =**	0.3707

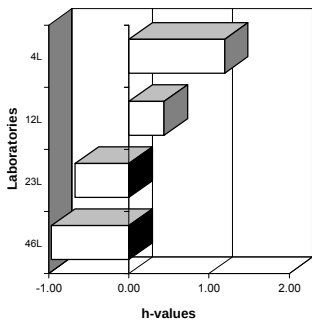
Remarks: **none**



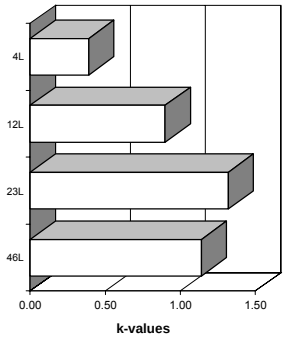
Sample: **Compost 1**

Element: **PCB 118**

Mandel's h statistics
(Compost 1 - PCB 118)



Mandel's k statistics
(Compost 1 - PCB 118)



Unit: ng/kg

Mandel's k statistics (Compost 1 - PCB 118)
Mandel's h statistics (Compost 1 - PCB 118)
Compost 1 - PCB 118 -- Mean PARM = 1.95 [ng/kg]

	General calc.parm.
T1=	3.11260E+01
T2=	6.13277E+01
T3=	16
T4=	64
T5=	4.3094E-01
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

End Result:

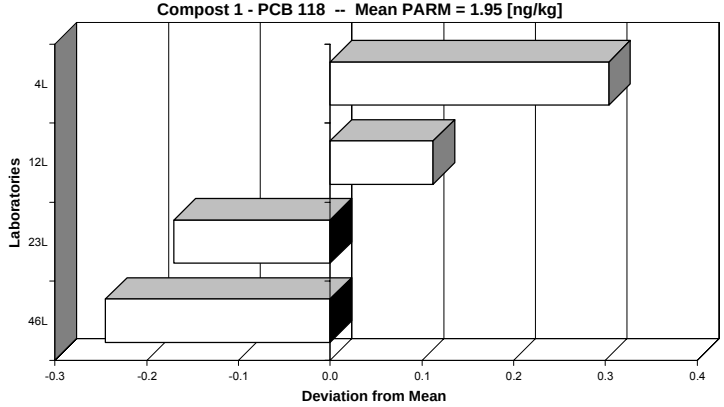
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
46L	1.7000	0.216	4		-0.96	1.14		Fail	1.7000	0.2160		4	3	-0.25
23L	1.7750	0.250	4		-0.67	1.32		Fail	1.7750	0.2500		4	3	-0.17
12L	2.0575	0.170	4		0.44	0.90			2.0575	0.1704		4	3	0.11
4L	2.2490	0.074	4		1.19	0.39	Fail		2.2490	0.0739		4	3	0.30
Tot.gem	1.945	0.178 ng/kg		*1%-level:*	1.49	(1.67)		4	*1.9454*	()		4	3	
Tot.std=	0.254	0.076		*5%-level:*	1.42	(1.5)								

RESULTS: **Mean =** **1.94538** *ng/kg*

Repeatability variance	**S2r =**	0.03591				
Repeatability std.	**Sr =**	**0.18950**	-->	9.74%	**r =**	0.5306
Between lab variance	**S2L =**	0.05569				
Reproducibility var.	**S2R =**	0.09160				
Reproducibility std.	**SR =**	**0.30265**	-->	15.56%	**R =**	0.8474

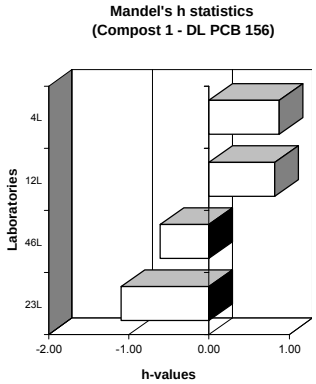
Remarks: **none**

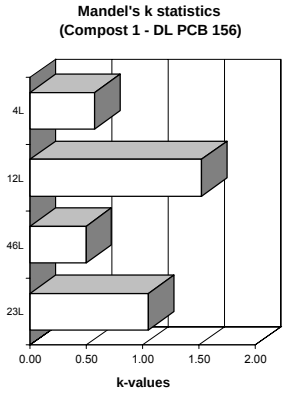
Compost 1 - PCB 118 -- Mean PARM = 1.95 [ng/kg]



Sample: **Compost 1**

Element: **DL PCB 156**





Unit: ng/kg

Mandel's k statistics (Compost 1 - DL PCB 156)
Mandel's h statistics (Compost 1 - DL PCB 156)

Compost 1 - DL PCB 156 -- Mean PARM = 0.882 [ng/kg]

General calc.parm.	
T1=	1.41164E+01
T2=	1.27037E+01
T3=	16
T4=	64
T5=	6.9603E-01
n=	variabel
p=	4
N-table=	*4*

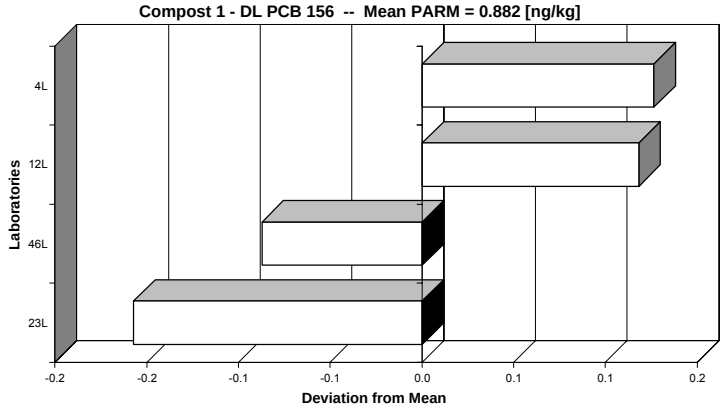
Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
23L	0.7250	0.254	4		-1.09	1.05		
46L	0.7950	0.120	4		-0.61	0.50		
12L	1.0005	0.366	4		0.82	1.52	!	
4L	1.0086	0.138	4		0.88	0.57		
Tot.gem	0.882	0.219	ng/kg	*1%-level:*	1.49	(1.67)		4
Tot.std=	0.144	0.115		*5%-level:*	1.42	(1.5)		

End Result:

PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
0.7250	0.2536		4	3	-0.16
0.7950	0.1196		4	3	-0.09
1.0005	0.3664		4	3	0.12
1.0086	0.1383		4	3	0.13
0.8823	()		4	3	

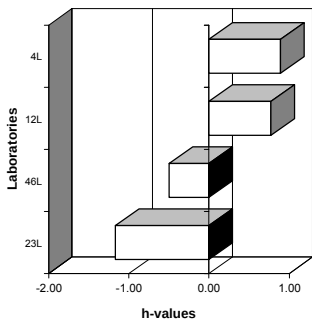
RESULTS:	**Mean =**	**0.88228**	*ng/kg*			
Repeatability variance	**S2r =**	0.05800				
Repeatability std.	**Sr =**	**0.24084**	-->	27.30%	**r =**	0.6743
Between lab variance	**S2L =**	0.00626				
Reproducibility var.	**S2R =**	0.06426				
Reproducibility std.	**SR =**	**0.25350**	-->	28.73%	**R =**	0.7098
Remarks:	**none**					



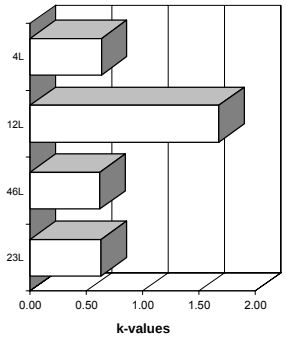
Sample: **Compost 1**

Element: **PCB 157**

Mandel's h statistics (Compost 1 - PCB 157)



Mandel's k statistics (Compost 1 - PCB 157)



Unit: ng/kg

Mandel's k statistics (Compost 1 - PCB 157)
Mandel's h statistics (Compost 1 - PCB 157)
Compost 1 - PCB 157 -- Mean PARM = 0.102 [ng/kg]

	General calc.parm.
T1=	1.63670E+00
T2=	1.70543E-01
T3=	16
T4=	64
T5=	3.7135E-03
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
23L	0.0835	0.011	4		-1.17	0.63		Fail	0.0835	0.0111		4	3	-0.02
46L	0.0943	0.011	4		-0.50	0.62			0.0943	0.0109		4	3	-0.01
12L	0.1148	0.030	4		0.77	1.68	!!		0.1148	0.0295		4	3	0.01
4L	0.1167	0.011	4		0.89	0.63			0.1167	0.0111		4	3	0.01
Tot.gem	0.102	0.016 ng/kg		*1%-level:*	1.49	(1.67)		4	*0.1023*	()		4	3	
Tot.std=	0.016	0.009		*5%-level:*	1.42	(1.5)								

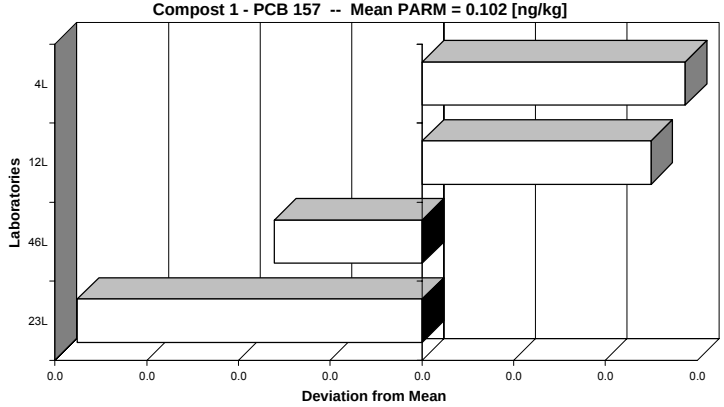
End Result:

RESULTS: **Mean =** **0.10229** *ng/kg*

Repeatability variance	**S2r =**	0.00031				
Repeatability std.	**Sr =**	**0.01759**	-->	17.20%	**r =**	0.0493
Between lab variance	**S2L =**	0.00018				
Reproducibility var.	**S2R =**	0.00049				
Reproducibility std.	**SR =**	**0.02218**	-->	21.68%	**R =**	0.0621

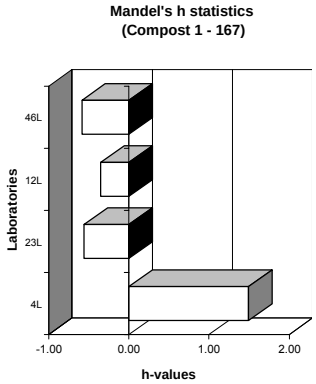
Remarks: **none**

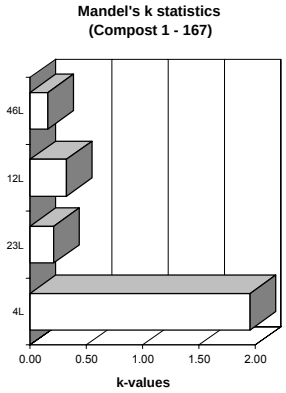
Compost 1 - PCB 157 -- Mean PARM = 0.102 [ng/kg]



Sample: **Compost 1**

Element: **167**





Unit: ng/kg

Mandel's k statistics (Compost 1 - 167)
Mandel's h statistics (Compost 1 - 167)

#NUM!

	General calc.parm.
T1=	#NUM!
T2=	#NUM!
T3=	
T4=	
T5=	
n=	variabel
p=	
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
4L	1.0995	0.941	4	!!	1.49	1.96	!!	
23L	0.4000	0.101	4		-0.56	0.21		
12L	0.4700	0.155	4		-0.35	0.32		
46L	0.3900	0.075	4		-0.59	0.16		
Tot.gem	0.590	0.318 ng/kg		*1%-level:*	1.49	(1.67)		
Tot.std=	0.342	0.417		*5%-level:*	1.42	(1.5)		

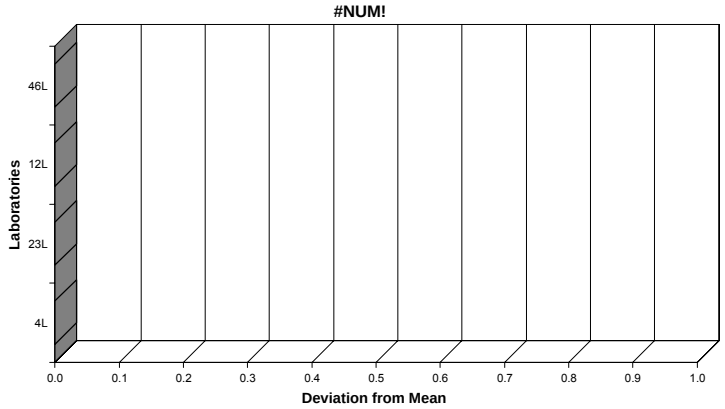
End Result:

PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
-	-	,4L	-	-	#NUM!
-	-	,23L	-	-	#NUM!
-	-	,12L	-	-	#NUM!
#NUM!	-	,46L	-	-	#NUM!
#NUM!		(4L,23L,12L,46L)		-1	

RESULTS: **Mean =** **#NUM!** *ng/kg*

Repeatability variance	**S2r =**	#DIV/0!				
Repeatability std.	**Sr =**	**#DIV/0!**	-->	#DIV/0!	**r =**	#DIV/0!
Between lab variance	**S2L =**	#NUM!				
Reproducibility var.	**S2R =**	#NUM!				
Reproducibility std.	**SR =**	**#NUM!**	-->	#NUM!	**R =**	#NUM!

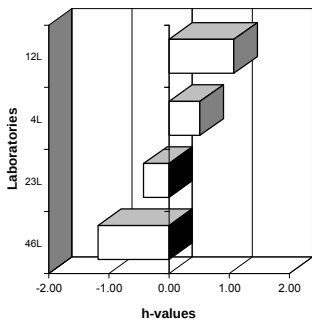
Remarks: **4 Labs rejected! (4L,23L,12L,46L)**



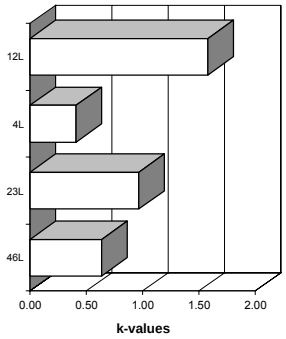
Sample: **Compost 1**

Element: **PCB 189**

Mandel's h statistics
(Compost 1 - PCB 189)



Mandel's k statistics
(Compost 1 - PCB 189)



Unit: ng/kg

Mandel's k statistics (Compost 1 - PCB 189)
Mandel's h statistics (Compost 1 - PCB 189)
Compost 1 - PCB 189 -- Mean PARM = 0.177 [ng/kg]

	General calc.parm.
T1=	2.82910E+00
T2=	5.03453E-01
T3=	16
T4=	64
T5=	5.2370E-02
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
46L	0.1575	0.042	4		-1.18	0.63		
23L	0.1700	0.064	4		-0.42	0.97		
4L	0.1853	0.027	4		0.52	0.41		
12L	0.1945	0.104	4		1.08	1.58	!	
Tot.gem	0.177	0.059 ng/kg		*1%-level:*	1.49	(1.67)		4
Tot.std=	0.016	0.034		*5%-level:*	1.42	(1.5)		

End Result:

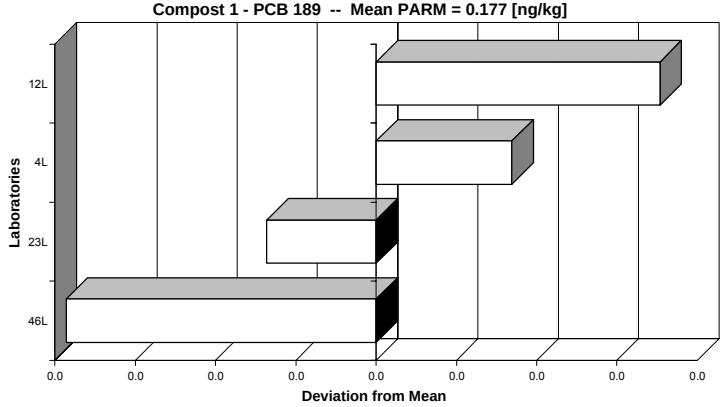
PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
0.1575	0.0419		4	3	-0.02
0.1700	0.0638		4	3	-0.01
0.1853	0.0271		4	3	0.01
0.1945	0.1044		4	3	0.02
0.1768	()		4	3	

RESULTS: **Mean =** **0.17682** *ng/kg*

Repeatability variance	**S2r =**	0.00436				
Repeatability std.	**Sr =**	**0.06606**	-->	37.36%	**r =**	0.1850
Between lab variance	**S2L =**	-0.00082				
Reproducibility var.	**S2R =**	0.00436				
Reproducibility std.	**SR =**	**0.06606**	-->	37.36%	**R =**	0.1850

Remarks: **none**

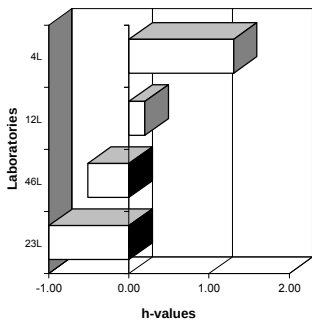
Compost 1 - PCB 189 -- Mean PARM = 0.177 [ng/kg]



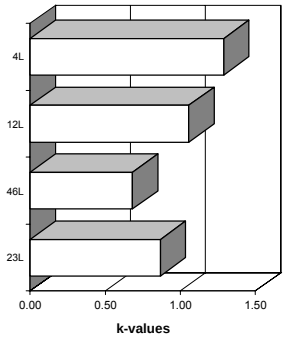
Sample: **Compost 1**

Element: **Total DL PCB**

Mandel's h statistics
(Compost 1 - Total DL PCB)



Mandel's k statistics
(Compost 1 - Total DL PCB)



Unit: ng/kg

Mandel's k statistics (Compost 1 - Total DL PCB)
Mandel's h statistics (Compost 1 - Total DL PCB)
Compost 1 - Total DL PCB -- Mean PARM = 4.66 [ng/kg]

	General calc.parm.
T1=	7.46177E+01
T2=	3.57297E+02
T3=	16
T4=	64
T5=	8.1859E+00
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
23L	3.7860	0.716	4		-1.00	0.87		Fail
46L	4.2123	0.564	4		-0.51	0.68		Fail
12L	4.8432	0.874	4		0.20	1.06		
4L	5.8130	1.065	4		1.30	1.29	Fail	
Tot.gem	4.664	0.805	ng/kg		*1%-level:*	1.49	(1.67)	
Tot.std=	0.881	0.215			*5%-level:*	1.42	(1.5)	

End Result:

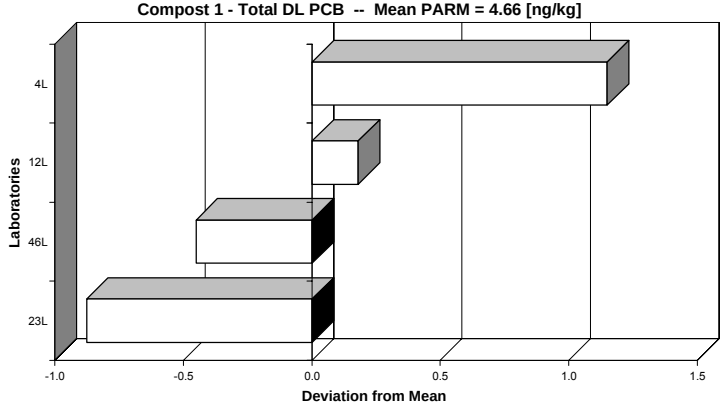
	PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
	3.7860	0.7161		4	3	-0.88
	4.2123	0.5635		4	3	-0.45
	4.8432	0.8741		4	3	0.18
	5.8130	1.0650		4	3	1.15
4	*4.6636*	()		4	3	

RESULTS: **Mean =** **4.66361** *ng/kg*

Repeatability variance	**S2r =**	0.68216				
Repeatability std.	**Sr =**	**0.82593**	-->	17.71%	**r =**	2.3126
Between lab variance	**S2L =**	0.60520				
Reproducibility var.	**S2R =**	1.28736				
Reproducibility std.	**SR =**	**1.13462**	-->	24.33%	**R =**	3.1769

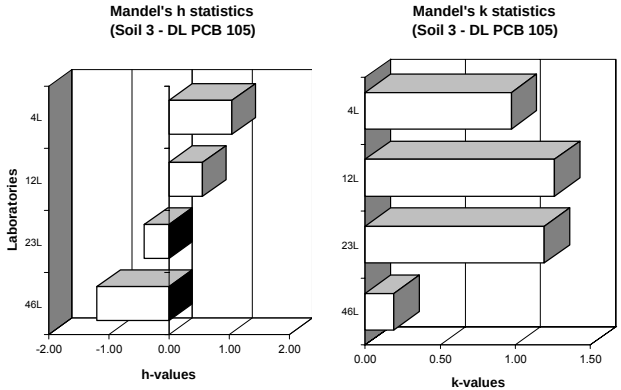
Remarks: **none**

Compost 1 - Total DL PCB -- Mean PARM = 4.66 [ng/kg]



Sample: **Soil 3**

Element: **DL PCB 105**



Unit: ng/kg

Mandel's k statistics (Soil 3 - DL PCB 105)
Mandel's h statistics (Soil 3 - DL PCB 105)

Soil 3 - DL PCB 105 -- Mean PARM = 0.096 [ng/kg]

General calc.parm.	
T1=	1.42530E+00
T2=	1.36772E-01
T3=	15
T4=	57
T5=	3.0222E-04
n=	variabel
p=	4
N-table=	*4*

Mandel's statistics

LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*
46L	0.0825	0.001	4		-1.20	0.19		Fail
23L	0.0913	0.006	4		-0.41	1.19		
12L	0.1020	0.007	4		0.56	1.26	Fail	
4L	0.1074	0.005	3		1.04	0.97	Fail	
Tot.gem	0.096	0.005	ng/kg		*1%-level:*	1.49	(1.67)	
Tot.std=	0.011	0.003			*5%-level:*	1.42	(1.5)	

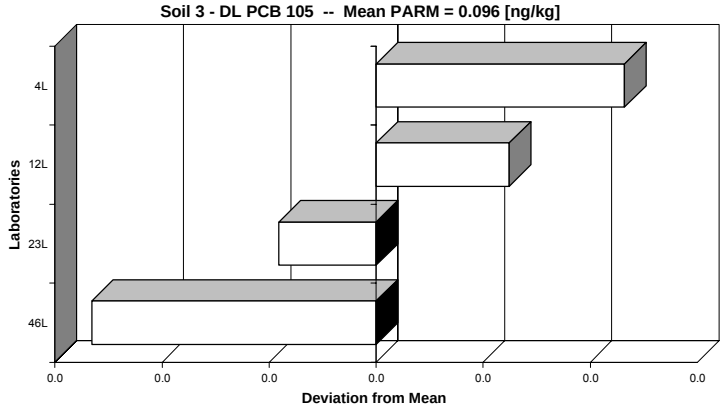
End Result:

	PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
	0.0825	0.0010		4	3	-0.01
	0.0913	0.0062		4	3	0.00
	0.1020	0.0066		4	3	0.01
	0.1074	0.0051		3	2	0.01
4	*0.0958*	()		4	3	

RESULTS: **Mean =** **0.09579** *ng/kg*

Repeatability variance	**S2r =**	0.00003				
Repeatability std.	**Sr =**	**0.00524**	-->	5.47%	**r =**	0.0147
Between lab variance	**S2L =**	0.00011				
Reproducibility var.	**S2R =**	0.00014				
Reproducibility std.	**SR =**	**0.01182**	-->	12.34%	**R =**	0.0331

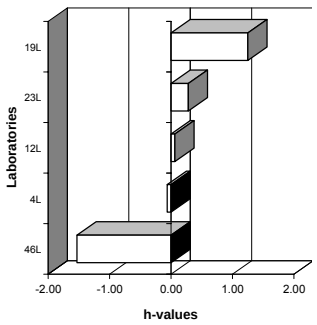
Remarks: **none**



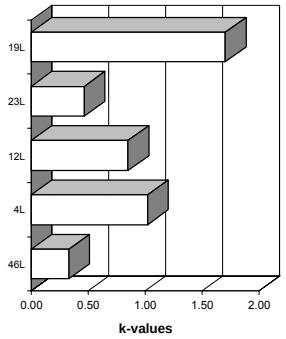
Sample: **Soil 3**

Element: **DL PCB 118**

Mandel's h statistics (Soil 3 - DL PCB 118)



Mandel's k statistics (Soil 3 - DL PCB 118)



Unit: ng/kg

Mandel's k statistics (Soil 3 - DL PCB 118)
Mandel's h statistics (Soil 3 - DL PCB 118)
Soil 3 - DL PCB 118 -- Mean PARM = 0.218 [ng/kg]

	General calc.parm.
T1=	4.09500E+00
T2=	9.07083E-01
T3=	19
T4=	73
T5=	3.7118E-03
n=	variabel
p=	5
N-table=	*4*

Mandel's statistics

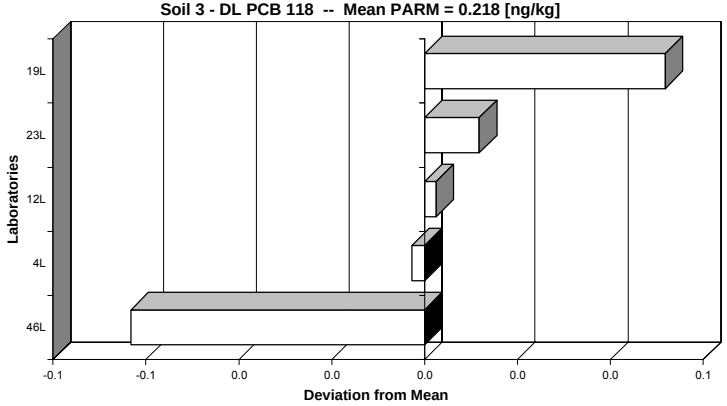
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark*	*\vX > AvST+2std*	*AvX < AvST-2std*
46L	0.1550	0.006	4		-1.53	0.33			Fail
4L	0.2155	0.018	4		-0.07	1.02			
12L	0.2208	0.015	4		0.06	0.85			
23L	0.2300	0.008	4		0.28	0.47			
19L	0.2700	0.030	3		1.25	1.70	!	Fail	
Tot.gem	0.218	0.015 ng/kg		*1%-level:*	1.72	(1.73)			
Tot.std=	0.041	0.009		*5%-level:*	1.57	(1.53)			

End Result:

	PARM	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
	0.1550	0.0058		4	3	-0.06
	0.2155	0.0179		4	3	0.00
	0.2208	0.0149		4	3	0.00
	0.2300	0.0082		4	3	0.01
	0.2700	0.0299		3	2	0.05
5	*0.2183*	()		5	4	

RESULTS:	**Mean =**	**0.21825**	*ng/kg*			
Repeatability variance	**S2r =**	0.00027				
Repeatability std.	**Sr =**	**0.01628**	-->	7.46%	**r =**	0.0456
Between lab variance	**S2L =**	0.00155				
Reproducibility var.	**S2R =**	0.00181				
Reproducibility std.	**SR =**	**0.04256**	-->	19.50%	**R =**	0.1192
Remarks:	**none**					

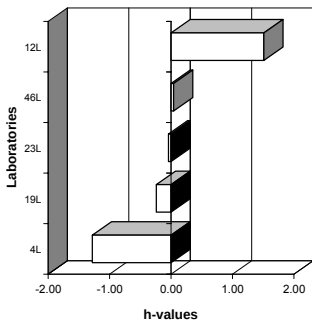
Soil 3 - DL PCB 118 -- Mean PARM = 0.218 [ng/kg]



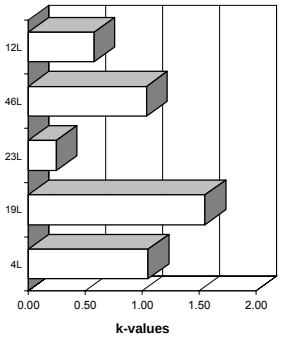
Sample: **Soil 3**

Element: **Total DL PCB**

Mandel's h statistics
(Soil 3 - Total DL PCB)



Mandel's k statistics
(Soil 3 - Total DL PCB)



Unit: ng/kg

Mandel's k statistics (Soil 3 - Total DL PCB)
Mandel's h statistics (Soil 3 - Total DL PCB)

Soil 3 - Total DL PCB -- Mean PARM = 0.37 [ng/kg]

General calc.parm.	
T1=	7.03950E+00
T2=	2.66077E+00
T3=	19
T4=	73
T5=	2.7290E-02
n=	variabel
p=	5
N-table=	*4*

Mandel's statistics

End Result:

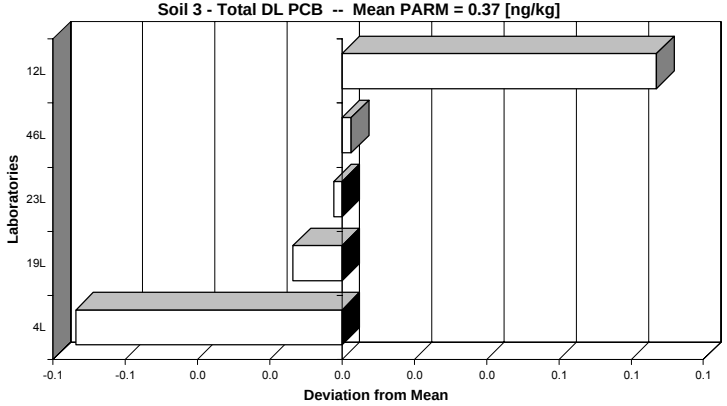
LAB	*PARM-gem*	*Stdev*	*N*	*h-mark*	*h*	*k*	*k-mark \vX > AvST+2std*	*AvX < AvST-2std*	*PARM*	*Stdev*	*Rej.labs*	*N*	*N-1*	*dev_mean*
4L	0.2961	0.049	4		-1.28	1.05		Fail	0.2961	0.0491		4	3	-0.07
19L	0.3562	0.072	3		-0.24	1.55			0.3562	0.0724		3	2	-0.01
23L	0.3675	0.012	4		-0.04	0.25			0.3675	0.0116		4	3	0.00
46L	0.3724	0.048	4		0.04	1.04			0.3724	0.0484		4	3	0.00
12L	0.4568	0.027	4		1.51	0.58	Fail		0.4568	0.0270		4	3	0.09
Tot.gem	0.370	0.042 ng/kg		*1%-level:*	1.72	(1.73)		5	*0.3698*	()		5	4	
Tot.std=	0.057	0.023		*5%-level:*	1.57	(1.53)								

RESULTS: **Mean =** **0.36979** *ng/kg*

Repeatability variance	**S2r =**	0.00195				
Repeatability std.	**Sr =**	**0.04415**	-->	11.94%	**r =**	0.1236
Between lab variance	**S2L =**	0.00296				
Reproducibility var.	**S2R =**	0.00491				
Reproducibility std.	**SR =**	**0.07005**	-->	18.94%	**R =**	0.1962

Remarks: **none**

Soil 3 - Total DL PCB -- Mean PARM = 0.37 [ng/kg]



Annex 4:

Raw data submitted

Sample:	Compost 1	
Element:	3,4,6,7,8-HpCDD	
LAB	PARM	[ng/kg]
4L	244.90	
4L	231.00	
4L	240.00	
4L	233.20	
23L	230.00	
23L	220.00	
23L	270.00	
23L	230.00	
12L	172.26	
12L	172.19	
12L	170.11	
12L	182.67	
56L	75.70	
56L	53.00	
56L	67.50	
56L	64.60	
46L	180.00	
46L	140.00	
46L	170.00	
46L	170.00	

Sample:	Compost 1	
Element:	?,3,4,7,8-HxCDD	
LAB	PARM	[ng/kg]
12L	0.77	
12L	0.84	
12L	0.88	
12L	0.67	
46L	0.85	
46L	0.48	
46L	0.72	
46L	0.69	

Sample:	Compost 1	
Element:	;3,6,7,8-HxCDD	
LAB	PARM	[ng/kg]
4L	5.10	
4L	4.10	
4L	4.00	
4L	5.20	
12L	3.29	
12L	3.20	
12L	3.00	
12L	3.32	
46L	3.40	
46L	2.40	
46L	3.10	
46L	3.20	

Sample:	Compost 1	
Element:	,3,7,8,9-HxCDD	
LAB	PARM	[ng/kg]
4L	2.60	
4L	2.10	
4L	2.30	
4L	2.40	
12L	1.53	
12L	1.49	
12L	1.49	
12L	1.67	
56L	2.70	
56L	3.10	
46L	1.60	
46L	1.20	
46L	1.60	
46L	1.80	

Sample:	Compost 1	
Element:	,2,3,7,8-PeCD	
LAB	PARM	[ng/kg]
12L	0.55	
12L	0.48	
12L	0.59	
12L	0.66	
46L	0.56	
46L	0.54	
46L	0.47	
46L	0.46	

Sample:	Compost 1	
Element:	2,3,7,8-TCDD	
LAB	PARM	[ng/kg]
12L	0.14	
12L	0.18	
12L	0.19	
12L	0.18	

Sample:	Compost 1	
Element:	OCDD	
LAB	PARM	[ng/kg]
4L	1502.90	
4L	1248.80	
4L	1377.20	
4L	1257.30	
23L	1700.00	
23L	1400.00	
23L	1600.00	
23L	1600.00	
12L	924.48	
12L	861.05	
12L	890.34	
12L	1149.65	
56L	564.70	
56L	453.40	
56L	569.30	
56L	453.70	
46L	880.00	
46L	710.00	
46L	620.00	
46L	850.00	

Sample:	Sludge 1	
Element:	3,4,6,7,8-HpCDD	
LAB	PARM	[ng/kg]
4L	236.70	
4L	232.50	
4L	246.10	
4L	231.60	
23L	160.00	
23L	160.00	
23L	180.00	
23L	210.00	
12L	170.66	
12L	170.64	
12L	162.66	
12L	162.18	
56L	170.10	
56L	115.60	
56L	109.50	
56L	178.00	
46L	190.00	
46L	200.00	
46L	210.00	
46L	210.00	
19L	174.00	
19L	208.00	
19L	150.00	
19L	208.00	

Sample:	Sludge 1	
Element:	?,3,4,7,8-HxCDD	
LAB	PARM	[ng/kg]
4L	4.60	
4L	4.00	
4L	4.00	
4L	3.80	
12L	2.87	
12L	2.97	
12L	3.32	
12L	3.13	
56L	16.50	
46L	3.30	
46L	3.40	
46L	3.60	
19L	3.42	
19L	3.53	
19L	3.10	
19L	3.49	

Sample:	Sludge 1	
Element:	?,3,6,7,8-HxCDD	
LAB	PARM	[ng/kg]
4L	10.90	
4L	11.90	
4L	11.80	
4L	10.00	
12L	7.14	
12L	7.47	
12L	7.73	
12L	7.64	
56L	6.60	
56L	5.30	
56L	8.70	
56L	9.80	
46L	8.40	
46L	7.50	
46L	8.10	
46L	8.80	
19L	7.72	
19L	7.69	
19L	5.40	
19L	6.97	

Sample:	Sludge 1	
Element:	2,3,7,8,9-HxCDD	
LAB	PARM	[ng/kg]
4L	6.00	
4L	7.60	
4L	6.70	
4L	5.90	
12L	4.04	
12L	3.81	
12L	3.80	
12L	4.08	
56L	10.50	
56L	8.90	
56L	12.40	
56L	10.80	
46L	4.90	
46L	4.90	
46L	3.30	
46L	5.20	
19L	13.00	
19L	4.40	
19L	16.30	
19L	4.35	

Sample:	Sludge 1	
Element:	2,3,7,8-PeCDD	
LAB	PARM	[ng/kg]
4L	3.60	
4L	4.90	
4L	4.20	
4L	3.50	
12L	1.66	
12L	1.95	
12L	1.53	
12L	1.84	
46L	2.10	
46L	1.70	
46L	1.70	
46L	1.70	
19L	1.66	
19L	1.62	
19L	0.94	
19L	1.97	

Sample:	Sludge 1	
Element:	2,3,7,8-TCDD	
LAB	PARM	[ng/kg]
12L	0.88	
12L	0.91	
12L	0.88	
12L	0.91	
46L	0.49	
46L	0.54	
46L	0.37	
19L	1.16	
19L	0.75	

Sample:	Sludge 1	
Element:	OCDD	
LAB	PARM	[ng/kg]
4L	1840.70	
4L	1823.30	
4L	1995.70	
4L	1923.90	
23L	1500.00	
23L	1700.00	
23L	2000.00	
23L	1800.00	
12L	1165.99	
12L	1167.30	
12L	1130.76	
12L	1132.28	
56L	1514.50	
56L	884.40	
56L	1029.50	
56L	1698.60	
46L	1400.00	
46L	1400.00	
46L	1400.00	
46L	1500.00	
19L	1230.00	
19L	1470.00	
19L	1150.00	
19L	1630.00	

Sample:	Soil 3	
Element:	3,4,6,7,8-HpCDD	
LAB	PARM	[ng/kg]
4L	7.50	
4L	9.30	
4L	7.50	
4L	8.00	
23L	10.00	
23L	8.50	
23L	7.30	
23L	12.00	
12L	5.63	
12L	5.33	
12L	5.61	
12L	5.55	
56L	13.90	
56L	10.50	
56L	16.60	
56L	9.70	
46L	6.50	
46L	5.90	
46L	5.90	
46L	5.70	
19L	7.10	
19L	6.07	
19L	6.47	

Sample:	Soil 3	
Element:	?,3,4,7,8-HxCDD	
LAB	PARM	[ng/kg]
12L	0.05	
12L	0.06	
12L	0.08	
12L	0.06	
19L	0.13	

Sample:	Soil 3	
Element:	?,3,6,7,8-HxCDD	
LAB	PARM	[ng/kg]
12L	0.29	
12L	0.33	
12L	0.34	
12L	0.32	
46L	0.29	
46L	0.31	
46L	0.23	
19L	0.53	

Sample:	Soil 3	
Element:	1,2,3,7,8,9-HxCDD	
LAB	PARM	[ng/kg]
12L	0.19	
12L	0.11	
12L	0.14	
12L	0.14	
56L	2.30	
46L	0.21	
46L	0.21	
46L	0.18	

Sample:	Spoil 3	
Element:	2,3,7,8-PeCDD	
LAB	PARM	[ng/kg]
12L	0.09	
12L	0.11	
12L	0.08	
12L	0.11	

Sample:	Soil 3	
Element:	2,3,7,8-TCDD	
LAB	PARM	[ng/kg]
19L	0.84	
19L	0.26	

Sample:	Soil 3	
Element:	OCDD	
LAB	PARM	[ng/kg]
4L	71.30	
4L	69.30	
4L	64.90	
4L	66.60	
23L	72.00	
23L	81.00	
23L	70.00	
23L	74.00	
12L	45.30	
12L	47.03	
12L	44.04	
12L	43.64	
56L	141.50	
56L	95.70	
56L	181.50	
56L	66.00	
46L	46.00	
46L	44.00	
46L	46.00	
46L	44.00	
19L	51.00	
19L	55.20	
19L	50.30	

Sample:	Compost 1	
Element:	3,4,6,7,8-HpCDF	
LAB	PARM	[ng/kg]
4L	28.80	
4L	21.20	
4L	18.90	
4L	19.10	
23L	17.00	
23L	25.00	
23L	12.00	
23L	39.00	
12L	14.04	
12L	12.20	
12L	12.78	
12L	14.10	
56L	9.56	
56L	7.86	
56L	7.30	
56L	8.83	
46L	12.00	
46L	10.00	
46L	11.00	
46L	13.00	

Sample:	Compost 1	
Element:	3,4,7,8,9-HpCDF	
LAB	PARM	[ng/kg]
4L	5.60	
4L	2.70	
4L	2.60	
12L	1.16	
12L	1.15	
12L	1.22	
12L	1.30	
56L	3.79	
46L	1.10	
46L	0.52	
46L	1.40	
46L	1.20	

Sample:	Compost 1	
Element:	,3,4,7,8-HxCDF	
LAB	PARM	[ng/kg]
4L	2.90	
4L	2.60	
4L	2.50	
4L	2.70	
12L	1.81	
12L	1.69	
12L	1.79	
12L	1.47	
56L	2.60	
56L	4.50	
56L	3.00	
56L	4.60	
46L	1.70	
46L	1.20	
46L	1.90	
46L	1.70	

Sample:	Compost 1	
Element:	,3,6,7,8-HxCDF	
LAB	PARM	[ng/kg]
4L	2.90	
4L	2.50	
4L	2.50	
4L	2.50	
23L	10.00	
12L	1.76	
12L	1.67	
12L	1.62	
12L	1.69	
46L	1.40	
46L	0.84	
46L	1.50	
46L	1.60	

Sample:	Compost 1	
Element:	,3,7,8,9-HxCDF	
LAB	PARM	[ng/kg]
12L	0.27	
12L	0.26	
12L	0.20	
12L	0.32	
46L	0.15	
46L	0.11	

Sample:	Compost 1	
Element:	2,3,7,8-PeCDF	
LAB	PARM	[ng/kg]
4L	2.30	
4L	2.30	
4L	2.20	
12L	1.26	
12L	1.29	
12L	1.31	
12L	1.47	
56L	1.60	
46L	0.97	
46L	0.66	
46L	1.40	
46L	1.10	

Sample:	Compost 1	
Element:	,4,6,7,8-HxCDF	
LAB	PARM	[ng/kg]
4L	3.70	
4L	3.30	
4L	3.20	
4L	3.30	
12L	1.32	
12L	1.86	
12L	1.84	
12L	1.83	
56L	2.60	
56L	3.40	
56L	3.50	
46L	1.40	
46L	1.10	
46L	1.40	
46L	1.50	

Sample:	Compost 1	
Element:	3,4,7,8-PeCDF	
LAB	PARM	[ng/kg]
4L	3.00	
4L	2.60	
4L	2.80	
4L	2.70	
12L	3.10	
12L	1.90	
56L	1.50	
56L	1.50	
56L	3.07	
56L	1.90	
46L	1.40	
46L	0.87	
46L	1.70	
46L	1.30	

Sample:	Compost 1	
Element:	2,3,7,8-TCDF	
LAB	PARM	[ng/kg]
4L	3.50	
4L	3.20	
4L	3.00	
4L	3.10	
12L	2.85	
12L	2.67	
12L	2.65	
12L	2.76	
56L	1.70	
56L	2.20	
56L	2.20	
46L	1.80	
46L	1.30	
46L	2.10	
46L	1.80	

Sample:	Compost 1	
Element:	OCDF	
LAB	PARM	[ng/kg]
4L	31.40	
4L	28.50	
4L	29.30	
4L	31.30	
23L	40.00	
23L	29.00	
23L	35.00	
23L	320.00	
12L	24.79	
12L	22.72	
12L	22.62	
12L	44.17	
56L	31.14	
56L	34.57	
56L	19.14	
56L	24.67	
46L	21.00	
46L	16.00	
46L	21.00	
46L	20.00	

Sample:	Sludge 1	
Element:	3,4,6,7,8-HpCDF	
LAB	PARM	[ng/kg]
4L	127.60	
4L	125.60	
4L	114.30	
4L	128.50	
23L	100.00	
23L	110.00	
23L	110.00	
23L	98.00	
12L	85.25	
12L	87.47	
12L	86.06	
12L	87.37	
56L	87.02	
56L	61.01	
56L	64.35	
56L	93.84	
46L	89.00	
46L	89.00	
46L	96.00	
46L	98.00	
19L	83.20	
19L	95.60	
19L	78.90	
19L	93.80	

Sample:	Sludge 1	
Element:	3,4,7,8,9-HpCDF	
LAB	PARM	[ng/kg]
4L	7.20	
4L	8.20	
4L	7.50	
4L	7.90	
12L	5.43	
12L	5.36	
12L	5.41	
12L	5.38	
56L	10.75	
56L	8.28	
56L	15.22	
56L	9.05	
46L	6.10	
46L	6.20	
46L	4.10	
46L	9.70	
19L	6.25	
19L	6.69	
19L	6.84	
19L	6.58	

Sample:	Sludge 1	
Element:	2,3,4,7,8-HxCDF	
LAB	PARM	[ng/kg]
4L	12.30	
4L	12.30	
4L	13.00	
4L	12.70	
12L	9.89	
12L	9.98	
12L	9.85	
12L	9.52	
56L	51.00	
56L	15.20	
56L	18.40	
56L	20.50	
46L	11.00	
46L	11.00	
46L	11.00	
46L	18.00	
19L	8.30	
19L	10.40	
19L	7.03	
19L	10.50	

Sample:	Sludge 1	
Element:	2,3,6,7,8-HxCDF	
LAB	PARM	[ng/kg]
4L	9.70	
4L	9.40	
4L	9.50	
4L	10.20	
12L	6.51	
12L	6.69	
12L	7.12	
12L	6.43	
56L	7.40	
56L	3.90	
56L	6.40	
56L	6.40	
46L	6.60	
46L	6.60	
46L	7.20	
46L	7.40	
19L	7.01	
19L	7.41	
19L	8.77	
19L	7.31	

Sample:	Sludge 1	
Element:	2,3,7,8,9-HxCDF	
LAB	PARM	[ng/kg]
4L	3.20	
4L	3.10	
4L	2.70	
4L	3.10	
12L	0.87	
12L	0.69	
12L	0.84	
12L	0.70	
56L	7.60	
56L	4.30	
46L	0.62	
19L	0.95	
19L	2.60	
19L	0.82	
19L	2.71	

Sample:	Sludge 1	
Element:	2,3,7,8-PeCDF	
LAB	PARM	[ng/kg]
4L	7.50	
4L	6.60	
4L	6.70	
4L	6.90	
12L	4.62	
12L	4.45	
12L	4.39	
12L	4.61	
56L	8.70	
56L	2.10	
56L	3.10	
56L	5.50	
46L	4.50	
46L	3.60	
46L	5.30	
19L	4.75	
19L	4.82	
19L	5.14	
19L	4.90	

Sample:	Sludge 1	
Element:	3,4,6,7,8-HxCDF	
LAB	PARM	[ng/kg]
4L	12.40	
4L	11.40	
4L	13.00	
4L	12.10	
12L	7.01	
12L	7.50	
12L	7.58	
12L	7.39	
56L	15.20	
56L	8.90	
56L	9.50	
46L	7.20	
46L	7.50	
46L	7.20	
46L	8.00	
19L	12.20	
19L	8.87	
19L	11.30	
19L	8.89	

Sample:	Sludge 1	
Element:	3,4,7,8-PeCDF	
LAB	PARM	[ng/kg]
4L	12.70	
4L	11.60	
4L	12.90	
4L	12.50	
12L	9.60	
12L	6.40	
12L	9.10	
12L	14.20	
56L	9.65	
56L	6.35	
56L	9.14	
56L	14.20	
46L	8.40	
46L	8.10	
46L	8.70	
46L	13.00	
19L	6.24	
19L	10.50	
19L	1.10	
19L	11.30	

Sample:	Sludge 1	
Element:	2,3,7,8-TCDF	
LAB	PARM	[ng/kg]
4L	10.60	
4L	12.00	
4L	11.90	
4L	12.30	
12L	10.40	
12L	11.26	
12L	10.50	
12L	9.77	
56L	21.10	
56L	10.90	
56L	11.70	
56L	19.80	
46L	9.30	
46L	8.60	
46L	8.80	
46L	10.00	
19L	8.45	
19L	8.99	
19L	8.09	
19L	9.56	

Sample:	Sludge 1	
Element:	OCDF	
LAB	PARM	[ng/kg]
4L	237.10	
4L	248.10	
4L	261.90	
4L	272.70	
23L	160.00	
23L	200.00	
23L	200.00	
23L	160.00	
12L	188.57	
12L	191.70	
12L	180.37	
12L	180.96	
56L	164.48	
56L	129.08	
56L	144.56	
56L	207.68	
46L	170.00	
46L	170.00	
46L	160.00	
46L	190.00	
19L	224.00	
19L	267.00	
19L	160.00	
19L	259.00	

Sample:	Soil 3	
Element:	3,4,6,7,8-HpCDF	
LAB	PARM	[ng/kg]
4L	14.30	
4L	16.50	
4L	12.70	
4L	12.30	
23L	15.00	
23L	12.00	
23L	13.00	
23L	18.00	
12L	12.55	
12L	11.89	
12L	11.53	
12L	12.56	
56L	7.69	
56L	13.28	
56L	18.33	
56L	11.29	
46L	8.60	
46L	11.00	
46L	11.00	
46L	11.00	
19L	13.20	
19L	10.60	
19L	8.94	

Sample:	Soil 3	
Element:	3,4,7,8,9-HpCDF	
LAB	PARM	[ng/kg]
12L	0.21	
12L	0.15	
12L	0.18	
12L	0.18	
46L	0.27	
46L	0.25	

Sample:	Soil 3	
Element:	,3,4,7,8-HxCDF	
LAB	PARM	[ng/kg]
12L	0.21	
12L	0.24	
12L	0.29	
56L	0.29	
56L	2.60	
46L	0.23	
46L	0.15	
46L	0.20	
19L	0.24	
19L	0.13	

Sample:	Soil 3	
Element:	,3,6,7,8-HxCDF	
LAB	PARM	[ng/kg]
12L	0.23	
12L	0.22	
12L	0.21	
12L	0.21	
46L	0.16	
46L	0.19	
46L	0.13	

Sample:	Soil 3	
Element:	,3,7,8,9-HxCDF	
LAB	PARM	[ng/kg]
12L	0.04	
12L	0.03	
12L	0.09	
12L	0.05	

Sample:	Soil 3	
Element:	2,3,7,8-PeCDF	
LAB	PARM	[ng/kg]
12L	0.15	
12L	0.18	
12L	0.16	
12L	0.19	
19L	0.18	

Sample:	Soil 3	
Element:	3,4,6,7,8-HxCDF	
LAB	PARM	[ng/kg]
12L	0.47	
12L	0.38	
12L	0.48	
12L	0.50	
56L	2.80	
56L	15.20	
46L	0.42	
46L	0.36	
46L	0.30	
19L	0.48	
19L	0.55	

Sample:	Soil 3	
Element:	3,4,7,8-PeCDF	
LAB	PARM	[ng/kg]
56L	1.50	
56L	1.50	
56L	1.50	
56L	1.50	
19L	0.18	

Sample:	Soil 3	
Element:	2,3,7,8-TCDF	
LAB	PARM	[ng/kg]
12L	0.34	
12L	0.31	
12L	0.37	
12L	0.33	
56L	1.20	
56L	1.30	
19L	0.23	

Sample:	Soil 3	
Element:	OCDF	
LAB	PARM	[ng/kg]
4L	43.80	
4L	53.20	
4L	46.40	
4L	48.90	
23L	51.00	
23L	50.00	
23L	42.00	
23L	57.00	
12L	37.42	
12L	35.26	
12L	35.06	
12L	40.62	
56L	58.23	
56L	55.23	
56L	81.70	
56L	42.36	
46L	30.00	
46L	31.00	
46L	31.00	
46L	30.00	
19L	49.80	
19L	45.90	
19L	41.40	

Unit ng/kg **DL PCB**

Sample:	Compost 1
Element:	77
LAB	PARM
4L	0.1495
4L	0.1530
4L	0.1480
4L	0.1510
23L	0.1300
23L	0.1100
23L	0.1300
23L	0.1100
12L	0.1280
12L	0.1250
12L	0.1250
12L	0.1250
46L	0.1400
46L	0.1100
46L	0.1300
46L	0.1400

Sample:	Compost 1
Element:	81
LAB	PARM
12L	0.0051
12L	0.0051
12L	0.0051
12L	0.0046
46L	0.0310
46L	0.0300
46L	0.0370
46L	0.0340

Sample:	Compost 1
Element:	105
LAB	PARM
4L	0.7074
4L	0.7893
4L	0.7619
4L	0.9047
23L	0.5600
23L	0.4500
23L	0.5400
23L	0.5000
12L	0.7330
12L	0.7080
12L	0.7080
12L	0.8010
46L	0.6700
46L	0.6100
46L	0.7700
46L	0.7100

Sample:	Compost 1
Element:	114
LAB	PARM
12L	0.0686
12L	0.0678
12L	0.0691
12L	0.1020
46L	0.0250
46L	0.0210
46L	0.0270
46L	0.0230

Sample:	Compost 1
Element:	118
LAB	PARM
4L	2.3080
4L	2.3140
4L	2.1650
4L	2.2090
23L	2.1000
23L	1.5000
23L	1.8000
23L	1.7000
12L	2.0800
12L	1.9300
12L	1.9300
12L	2.2900
46L	1.6000
46L	1.5000
46L	2.0000
46L	1.7000

Sample:	Compost 1
Element:	123
LAB	PARM
4L	0.1967
4L	0.1952
4L	0.2178
4L	0.2413
12L	0.0265
12L	0.0254
12L	0.0253
12L	0.0253
46L	0.1400
46L	0.1300
46L	0.1900
46L	0.1600

Sample:	Compost 1
Element:	126
LAB	PARM
12L	0.0311
12L	0.0315
12L	0.0317
12L	0.0310
46L	0.0440
46L	0.0330
46L	0.0470
46L	0.0380

Sample:	Compost 1
Element:	156
LAB	PARM
4L	1.1580
4L	1.0920
4L	0.8698
4L	0.9146
23L	1.1000
23L	0.5400
23L	0.6300
23L	0.6300
12L	0.8300
12L	0.8090
12L	0.8130
12L	1.5500
46L	0.7600
46L	0.7000
46L	0.9700
46L	0.7500

Sample:	Compost 1
Element:	157
LAB	PARM
4L	0.1255
4L	0.1242
4L	0.1013
4L	0.1156
23L	0.1000
23L	0.0800
23L	0.0760
23L	0.0780
12L	0.1020
12L	0.0993
12L	0.0988
12L	0.1590
46L	0.0920
46L	0.0850
46L	0.1100
46L	0.0900

Sample:	Compost 1
Element:	167
LAB	PARM
4L	2.5050
4L	0.7580
4L	0.5583
4L	0.5767
23L	0.5500
23L	0.3300
23L	0.3500
23L	0.3700
12L	0.3970
12L	0.3900
12L	0.3900
12L	0.7030
46L	0.3700
46L	0.3300
46L	0.5000
46L	0.3600

Sample:	Compost 1
Element:	169
LAB	PARM
12L	0.0038
12L	0.0038
12L	0.0041
12L	0.0039
46L	0.0026
46L	0.0028
46L	0.0038
46L	0.0028

Sample:	Compost 1
Element:	189
LAB	PARM
4L	0.2089
4L	0.2069
4L	0.1541
4L	0.1712
23L	0.2600
23L	0.1200
23L	0.1700
23L	0.1300
12L	0.1450
12L	0.1370
12L	0.1450
12L	0.3510
46L	0.1400
46L	0.1300
46L	0.2200
46L	0.1400

Unit ng/kg **DL PCB**

Sample:	Sludge 1
Element:	77
LAB	PARM
4L	7.4230
4L	6.9340
4L	7.6600
4L	7.6320
23L	5.7000
23L	5.2000
23L	5.7000
23L	5.7000
12L	6.4500
12L	6.6100
12L	6.2700
12L	6.2800
46L	6.9000
46L	7.0000
46L	7.3000
46L	6.7000
19L	7.5100
19L	7.1000
19L	6.2900

Sample:	Sludge 1
Element:	81
LAB	PARM
4L	0.7550
4L	0.6950
4L	0.7185
4L	0.7298
23L	0.3800
23L	0.4000
23L	0.4000
23L	0.3300
12L	0.3130
12L	0.3120
12L	0.3010
12L	0.3070
46L	0.8900
46L	0.7800
46L	0.9500
46L	0.7700
19L	0.4040
19L	0.2650
19L	0.4190

Sample:	Sludge 1
Element:	105
LAB	PARM
4L	23.0150
4L	21.5180
4L	20.2860
4L	24.3290
23L	16.0000
23L	16.0000
23L	16.0000
23L	17.0000
12L	18.3000
12L	18.2000
12L	18.3000
12L	18.0000
46L	19.0000
46L	19.0000
46L	23.0000
46L	19.0000
19L	24.2000
19L	14.5000

Sample:	Sludge 1
Element:	114
LAB	PARM
4L	1.6650
4L	1.8480
4L	1.7440
4L	1.6620
23L	0.9800
23L	1.0000
23L	0.9800
23L	1.0000
12L	1.6600
12L	1.7300
12L	1.6600
12L	1.6700
46L	1.0000
46L	1.1000
46L	1.2000
46L	1.0000
19L	1.1900
19L	1.4100
19L	0.9400
19L	1.8400

Sample:	Sludge 1
Element:	118
LAB	PARM
4L	37.5510
4L	38.1470
4L	37.9800
4L	35.6870
23L	31.0000
23L	32.0000
23L	31.0000
23L	33.0000
12L	33.4000
12L	33.9000
12L	33.1000
12L	32.3000
46L	29.0000
46L	31.0000
46L	55.0000
46L	30.0000
19L	28.0000
19L	27.6000
19L	28.7000
19L	28.0000

Sample:	Sludge 1
Element:	123
LAB	PARM
4L	5.2690
4L	4.2430
4L	4.8370
4L	4.4220
23L	0.7700
23L	0.8200
23L	0.6800
23L	0.7600
12L	0.8390
12L	0.8550
12L	0.8660
12L	0.8390
46L	1.7000
46L	1.6000
46L	5.2000
46L	1.8000
19L	0.7550
19L	0.8220
19L	0.7420
19L	0.8820

Sample:	Sludge 1
Element:	126
LAB	PARM
4L	0.2905
4L	0.2836
4L	0.2080
4L	0.2980
23L	0.1700
23L	0.1600
23L	0.1000
23L	0.1500
12L	0.2870
12L	0.2480
12L	0.2450
12L	0.2430
46L	0.3600
46L	0.3700
46L	0.3600
46L	0.3400
19L	0.1920
19L	0.2290
19L	0.2450

Sample:	Sludge1
Element:	156
LAB	PARM
4L	7.9900
4L	7.9280
4L	8.5850
4L	8.5120
23L	6.6000
23L	6.6000
23L	6.8000
23L	6.4000
12L	7.4500
12L	7.3200
12L	6.9900
12L	6.9900
46L	6.9000
46L	7.3000
46L	28.0000
46L	7.4000
19L	6.2200
19L	5.5500
19L	7.1100
19L	4.9500

Sample:	Sludge 1
Element:	157
LAB	PARM
4L	1.0654
4L	1.0352
4L	1.0913
4L	1.0472
23L	0.6600
23L	0.7300
23L	0.7200
23L	0.7600
12L	0.9650
12L	0.9170
12L	0.9130
12L	0.9110
46L	0.8500
46L	0.8700
46L	2.5000
46L	0.9000
19L	0.8850
19L	0.7020
19L	0.6570
19L	0.7600

Sample:	Sludge 1
Element:	167
LAB	PARM
4L	4.6320
4L	5.0117
4L	4.6360
4L	6.2340
23L	3.3000
23L	3.2000
23L	3.1000
23L	3.3000
12L	3.3100
12L	3.2000
12L	3.1500
12L	3.0500
46L	3.0000
46L	3.2000
46L	12.0000
46L	3.1000
19L	3.4000
19L	2.8200
19L	3.2800
19L	3.0800

Sample:	Sludge 1
Element:	169
LAB	PARM
4L	0.1008
4L	0.1089
12L	0.0273
12L	0.0245
12L	0.0237
12L	0.0234
46L	0.0190
46L	0.0190
46L	0.0200
46L	0.0230
19L	0.0799
19L	0.0280

Sample:	Sludge 1
Element:	189
LAB	PARM
4L	1.1490
4L	1.1580
4L	0.1320
4L	1.1245
23L	0.9200
23L	0.8400
23L	0.9200
23L	0.8800
12L	1.1800
12L	1.1100
12L	1.0900
12L	1.0900
46L	1.1000
46L	1.2000
46L	4.3000
46L	1.2000
19L	0.9990
19L	0.8780
19L	0.8390
19L	1.0200

UNIT ng/kg **DL PCB**

Sample:	Soil 3
Element:	77
LAB	PARM
12L	0.0261
12L	0.0248
12L	0.0252
12L	0.0242
46L	0.0240
46L	0.0220
46L	0.0260
46L	0.0230
19L	0.0330
19L	0.0200
19L	0.0350

Sample:	Soil 3
Element:	81
LAB	PARM
12L	0.0010
12L	0.0009
12L	0.0008
12L	0.0009
46L	0.0043
46L	0.0035
46L	0.0032
46L	0.0030

Sample:	Soil 3
Element:	105
LAB	PARM
4L	0.1115
4L	0.1017
4L	0.1090
23L	0.0850
23L	0.0870
23L	0.0980
23L	0.0950
12L	0.1110
12L	0.0951
12L	0.1010
12L	0.1010
46L	0.0830
46L	0.0810
46L	0.0830
46L	0.0830

Sample:	Soil 3
Element:	114
LAB	PARM
12L	0.0109
12L	0.0090
12L	0.0100
12L	0.0093
46L	0.0040
46L	0.0039
46L	0.0043
46L	0.0039
19L	0.1000
19L	0.1000

Sample:	Soil 3
Element:	118
LAB	PARM
4L	0.1920
4L	0.2330
4L	0.2120
4L	0.2250
23L	0.2200
23L	0.2400
23L	0.2300
23L	0.2300
12L	0.2420
12L	0.2090
12L	0.2200
12L	0.2120
46L	0.1500
46L	0.1500
46L	0.1600
46L	0.1600
19L	0.2920
19L	0.2360
19L	0.2820

Sample:	Soil 3
Element:	123
LAB	PARM
12L	0.0056
12L	0.0056
12L	0.0053
12L	0.0048
46L	0.0110
46L	0.0093
46L	0.0120
46L	0.0110

Sample:	Soil 3
Element:	126
LAB	PARM
12L	0.0020
12L	0.0019
12L	0.0020
12L	0.0021
46L	0.0037
46L	0.0022
46L	0.0031
46L	0.0019
19L	0.0016
19L	0.0041

Sample:	Soil 3
Element:	156
LAB	PARM
23L	0.0460
23L	0.0490
23L	0.0470
23L	0.0430
12L	0.0560
12L	0.0466
12L	0.0496
12L	0.0506
46L	0.0390
46L	0.0420
46L	0.0380
46L	0.0370

Sample:	Soil 3
Element:	157
LAB	PARM
12L	0.0104
12L	0.0083
12L	0.0087
12L	0.0090
46L	0.0065
46L	0.0069
46L	0.0069
46L	0.0064

Sample:	Soil 3
Element:	167
LAB	PARM
12L	0.0224
12L	0.0243
12L	0.0207
12L	0.0216
46L	0.0170
46L	0.0160
46L	0.0180
46L	0.0160

Sample:	Soil 3
Element:	169
LAB	PARM
12L	0.0005
12L	0.0005
12L	0.0005
12L	0.0005
46L	0.0004
46L	0.0003
46L	0.0826

Sample:	Soil 3
Element:	189
LAB	PARM
12L	0.0083
12L	0.0095
12L	0.0074
12L	0.0086
46L	0.0065
46L	0.0066
46L	0.0076
46L	0.0064

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Abstract

Project HORIZONTAL is interdisciplinary aiming at a harmonisation and horizontal standardisation of test procedures, in particular for sludge, soils and biowastes. In the context of this standardization project, a series of draft technical specification were designed upon an extensive desk study, fine-tuned after expert consultations and finally validated in international intercomparisons exercise.
This report summarises the work performed within the validation study of the draft standard for the determination of polychlorinated dibenzodioxins (PCDD), polychlorinated dibenzofurans (PCDF) and dioxins-like polychlorinated biphenyls (DL PCB) in soils, sludge and treated bio-waste using high resolution mass selective detection (HR GC-MS). It further explains the underlying statistical concept for the calculation of reproducibility and repeatability from intercomparisons data. In addition all single values, results of the statistical evaluation as well as background information on the validation materials used are described and explained.

The mission of the JRC is to provide customer-driven scientific and technical support for the conception, development, implementation and monitoring of EU policies. As a service of the European Commission, the JRC functions as a reference centre of science and technology for the Union. Close to the policy-making process, it serves the common interest of the Member States, while being independent of special interests, whether private or national.

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